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LONG SHELF LIFE
ORGANIC ELECTROLYTE BATTERY

FINAL REPORT
BY
L.F.ATHEARN and A.N.DEY
D A PROJECT NUMBER 1X6 28012 D628
JUNE 1971

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13. ABSTRACT The fabrication of prototype organic electrolyte cells having wide operating temperature range and high energy density has been completed. Seventy of these cells were assembled and delivered for evaluation tests. In tests of samples of these cells carried out at this laboratory, energy densities of 95 watt hours per pound at room temperature and 55 watt hours per pound at -30°C were obtained. Similar cell components were assembled to make fifteen 36 volt batteries with a diameter of 2.75 in. and a height of 5.0 inches. These batteries weighed 80-90% of the maximum weight (1.8 lbs) specified. The discharge of a prototype battery at room temperature using the specified discharge regime was performed. This test is continuing after 2350 hours and the battery is still above the 20 volt cutoff voltage. The required service on this test is 912 hours. Improved cells of the type used to make the 36 volt batteries were tested at room temperature on the specified loads. These cells are still running after 3880 hours of discharge and the cells are still above the 1.8 volt cutoff voltage. The service required on this test was also 912 hours. Other cells of the type used to make the 36 volt batteries were discharged at -30°C on the revised test schedule and they discharged for 592 hours before reaching the 1.8 volt cutoff voltage. The required service on this test was 480 hours. None of the improved cells were incorporated in the delivered end items because of the contractual limitations on the delivery schedule. The contractual objective for charge retention, high efficiency performance following three months storage at 55°C, was not attained. Reduced temperature storage is recommended for the cells and batteries. Some redesign of cell and battery structures is required to prevent electrolyte leakage and internal corrosion observed in the delivered end items.		

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**LONG SHELF LIFE ORGANIC ELECTROLYTE BATTERY
FINAL REPORT**

Report No. 4

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Prepared by

L.F. Athearn and A.N. Dey

P.R. MALLORY & CO. INC.
Laboratory for Physical Science
Northwest Industrial Park
Burlington, Massachusetts 01803

For

U.S. ARMY ELECTRONICS COMMAND, Fort Monmouth, New Jersey 07703

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ABSTRACT

The fabrication of prototype organic electrolyte cells having wide operating temperature range and high energy density has been completed. Seventy of these cells were assembled and delivered for evaluation tests. In tests of samples of these cells carried out at this laboratory, energy densities of 95 watt hours per pound at room temperature and 55 watt hours per pound at -30°C were obtained.

Similar cell components were assembled to make fifteen 36 volt batteries with a diameter of 2.75 in. and a height of 5.0 inches. These batteries weighed 80-90% of the maximum weight (1.8 lbs.) specified.

The discharge of a prototype battery at room temperature using the specified discharge regime was performed. This test is continuing after 2350 hours and the battery is still above the 20 volt cutoff voltage. The required service on this test is 912 hours.

Improved cells of the type used to make the 36 volt batteries were tested at room temperature on the specified loads. These cells are still running after 3880 hours of discharge and the cells are still above the 1.8 volt cutoff voltage. The service required on this test was also 912 hours.

Other cells of the type used to make the 36 volt batteries were discharged at -30°C on the revised test schedule and they discharged for 592 hours before reaching the 1.8 volt cutoff voltage. The required service on this test was 480 hours.

None of the improved cells were incorporated in the delivered end items because of the contractual limitations on the delivery schedule.

The contractual objective of charge retention, high efficiency performance following three months storage at 55°C , was not attained. Reduced temperature storage is recommended for the cells and batteries. Some redesign of cell and battery structures is required to prevent electrolyte leakage and internal corrosion observed in the delivered end items.

1.0 INTRODUCTION

1.1 Background

The development of high energy density non-reserve type primary organic electrolyte systems has been the goal of many research and development efforts. Extensive studies of these systems in our laboratories during the past six years have led to the development of several non-reserve type primary systems which have very low intrinsic selfdischarge rates. Completely finished batteries have been made with a practical energy density of about 100 whrs/lb. The available shelf life data indicated that our systems had a selfdischarge rate below 3% capacity loss per year at room temperature.

The basic developments which made this exceptional performance possible were

- a) non-gassing systems,
- b) extremely low cathode material solubility,
- c) elimination of corrosion reactions between the electrolytes and the lithium anode, and
- d) a simplified low cost hermetically sealed package.

Because of our previous work in the organic field and the necessity of providing a cathode operative in the range from -20°F (-29°C) to 130°F (55°C), it was determined that a productive effort should be made to obtain a cathode suitable for these temperatures. Accordingly, proprietary cathodes were evaluated upon which Mallory had filed patent applications. These cathodes seemed to offer feasible systems in conjunction with light metal electrodes. The charts hereto appended indicate some of the results obtained in the use of these cathodes.

The U.S. Army Electronics Command expressed interest in the development of organic electrolyte cells which could be assembled into batteries for special applications. Preliminary estimates based on available cell performance data indicated that the energy density, low temperature operation, and long active shelf life were feasible. This information was the basis for the project.

1.2 Scope

The objectives of this project were to develop and make 100 organic electrolyte cells that would operate over the extended temperature range required for special military applications, deliver 100 watt hours per pound and have a high temperature storage capability. The details of this work statement are given in Appendix I.

When it was determined that cells could be made to deliver the energy density required under many of the conditions indicated, the project objectives were modified to replace 30 of the original 100 cells with fifteen 36 volt batteries. The revised objectives and requirements were:

- 1) Fifteen 36 volt batteries to be supplied in addition to seventy 3 volt cells.
- 2) Batteries to be $2.75 \pm 0.01 - 0.05 \times 5''$ maximum height with snap terminals on the cover.
- 3) Voltage: nominal 36 volts.
- 4) Electrical performance
 - a. constant 15,000 ohm load
 - b. 65 millisecond pulse load on 57 ohm load, 1200 times per day for 38 days
 - c. cutoff: 20 volts
- 5) Room temperature tests only.
- 6) Maximum battery weight 1.8 pounds.

1.3 Program

On the basis of the original work statement the project was planned and the program schedule indicated in Fig. 1 was adopted.

The cell design calculations and the shape restrictions required that the cell electrodes be 2.4 inches in diameter \times 0.040 to 0.050 thick. The cells that had been made for performance studies had 1.6" \times 0.96" \times 0.40" rectangular electrodes. Therefore new tooling was designed and built for the 2.4" diameter electrodes. During this tooling up period rectangular cells were made and tested on loads that produced the same cathode current densities as were required in the final cells.

The cell cans and covers were designed to be cold welded in a hydraulic operated die set. The resulting cell containers were checked and found to be hermetic.

When the tooling for the 2.4" electrodes was operational single cathode cells were made to evaluate their performance under the various test conditions. These cells were also used to study various electrolytes and cathode fabrication methods.

The initial LB-35 cells were assembled using the improvements found in the single cathode tests. The testing of these cells under the conditions required yielded lower than expected energy densities. The LB-35 cells were redesigned using more electrodes to reduce the current densities. These redesigned cells yielded the higher energy densities expected (90-95 whrs/lb at room temperature and 50-55 whrs/lb at -30°C). Seventy of these cells were constructed and delivered.

Following the revision of the project objectives a 36 volt battery was designed using the electrode structures and materials from the LB-35 cells. Fifteen of these batteries were constructed for delivery. These batteries consisted of eleven series connected 3 cathode cells potted in a metal cylindrical case. The final delivered batteries weighed approximately 90% of the maximum allowed (1.8 lb) and delivered approximately twice the hours service required.

The construction of these 15 batteries was carried out during an extension of the contract.

2.0 TOOLING AND EQUIPMENT

The size and shape of the cells that was specified required that new but standard tools be made for the parts fabrication. The use of lithium anodes in these cells required that some of the assembly steps be carried out in a controlled atmosphere.

2.1 Tooling for the 35AH Cells

2.1.1 Cathode Molding Dies. The cathodes used in these cells were molded in the die set shown in Fig. 2. These dies were fitted to a double acting hydraulic press. The cathodes were molded using two charges of cathode mix and an expanded metal grid. The grid was exposed after molding and a tab attached as indicated in Fig. 3.

2.1.2 Lithium Anode Tools. The anodes are made by cutting a 2-1/4" diameter disc from 0.020 lithium foil using an arch punch. This disc is

then laid on the stainless current collector Fig. 4 and pressed at approximately 2 tons. The resulting anode collector assembly is approximately 2-1/4" diameter x 0.018" to 0.019" thick.

2. 1. 3 Separator and Collector Dies. The separators and current collectors are cut from stock using steel rule dies. The separators are cut (2-27/64" diameter) from sheet filter paper stock by placing several layers on a cardboard sheet and pressing the steel rule die through the filter paper using a bench arbor press.

The collectors are cut from suitable nickel and stainless expanded metal (2-1/4" diameter). This operation is performed by placing several layers of expanded metal separated by sheets of paper on a hard wood block and positioning the steel rule die. The assembly is then pressed in a bench hydraulic press.

2. 1. 4 Stacking Jig. The cell stacks were assembled using the jig shown in Fig. 5. The electrodes were laid in with separators so that anode tabs projected from one side of the jig and the cathode tabs from the other. When the stack was complete it was removed from the jig, tied, and the tabs welded.

2. 1. 5 Cold Welding Tools. The hydraulic press and cold welding dies were assembled in a new dry box section attached to the present dry box. The cold welding tools consisted of two pairs of dies in one die set. The first pair made a compression weld between the glass to metal seal flange and the cover. These dies are easily removed from the die set leaving the extrusion weld dies in place for the final can closing. See Fig. 6 and Fig. 7.

The hydraulic press operating these dies is capable of 9 tons of force. During the compression weld a positive stop in the die set prevented over pressure on the cover and seal. In the case of the extrusion weld, the can containing the cell assembly was pushed down through the female portion of the dies by the upper or male portion. When the extrusion weld was made, a force of 2-1/2 to 4 tons was required.

Leak tests were performed on empty cans made by compression welding the seal to the cover and then extrusion welding the cover assembly to the can. These were helium leak tested using a test fixture shown in Fig. 8 and mass spectrometer type leak testing equipment.

2.2 Equipment for the 35AH Cells

2.2.1 Dry Box. The dry box and atmosphere purification equipment was supplied by Vacuum Atmospheres Corporation. The existing box is DriLab model HE-453-2. The new dry box section model HE-243 was added to the end of the existing equipment. A door between the sections permitted isolation of the cold welding area to facilitate equipment changes or repairs without disturbing the remainder of the cell construction operations.

Pressure control and atmosphere circulation connections were made to the added section to insure uniform atmosphere composition and separate purge capabilities. The added section has special high pressure hydraulic feedthroughs for the connection to the hydraulic cold welding press.

2.2.2 Atmosphere Purification System. The argon atmosphere was introduced to the dry box by means of an initial purge to remove air. Further purification was accomplished using Vacuum Atmospheres Dri-Train HE-373 BIN. This system was capable of removing water, oxygen and nitrogen to very low levels. This system was run continuously after the initial purge and maintained the moisture level at 1-2 ppm or -75°C dew point.

2.2.3 Moisture Measurement. The moisture level in the dry box was monitored daily with a Panametrics model 1000 hygrometer. This unit measures variations in an alumina capacitor probe threaded into a port of the dry box. Leaks or failures of the moisture removal system would be indicated by a rising dew point of the atmosphere. No such system was used to monitor the oxygen or nitrogen levels in the box atmosphere.

2.2.4 Molding Press. A large hydraulic press (150 ton capacity) was made available for this project and modified to operate the double acting cathode die set. The press was electrically powered, manually operated and controlled to provide a wide range of pressures, closing and ejection rates and holding times.

2.2.5 Cell Discharge Test Equipment. The pulse discharge test equipment was constructed to cycle test 24 cells on the load profile of continuous 15 milliwatt drain with a 6 second, 5 watt pulse repeated every ten minutes. The timing cycle was determined by an electromechanical timer and a time delay relay. Test relays controlled by the cycle timer switched the current

levels on constant current generators, see Block diagram Fig. 9. The generators were adjusted for the power levels as determined from Tables 1 through 6. (Appendix III.)

The cells were connected to the constant current generators and the voltage sensing circuit. The voltage sensing circuit maintained power on a running time meter until the cell voltage dropped below 1.5 volts, or other adjustable cutoff voltage. Cell voltages were determined by connecting a recorder directly across the cell terminals.

The complete circuit diagram and printed circuit board layout for this test equipment are shown in Figs. 10 and 11.

2.3 Tooling for the 36 Volt Batteries

The 36 volt batteries used the same electrodes and other cell components as the 35AH cells, therefore no new tooling was required.

2.4 Equipment for the 36 Volt Batteries

The 36 volt batteries were assembled using the same equipment and facilities as the 35AH cells. The testing of these batteries and the cells that comprised them was carried out using the circuits shown in Figs. 11 and 12.

3.0 CELL AND BATTERY DESIGNS

The final cell and battery designs were based on data obtained on single cathode cells in both rectangular and circular configurations. The rectangular cells (Fig. 15) were made only until the tooling for the circular cells was available and the results are given in quarterly reports 1, 2, and 3.

The circular cells (1 cathode, 2 anodes) were built (see Fig. 3) using various electrolytes in order to obtain the best performance at -30°C after $+55^{\circ}\text{C}$ storage.

3.1 35AH Cells

The initial 35AH cells were assembled from 13 type B cathodes and 14 pressed lithium foil anodes (Figs. 3 and 16). The electrodes were stacked with separators using the stacking jig shown in Fig. 5. The electrode stack was placed in a plastic bag and inserted in the metal can. Terminal connections were then welded to the anode and cathode tabs and the electrolyte added. The plastic bag was then sealed around the terminal tabs, potting compound was added, and the can cold welded in the press shown in Fig. 7.

During assembly and testing of these cells some deficiencies were noted that suggested design changes. The most important of these changes were:

- 1) an increase in the number of cathodes and anodes to 20 and 21, respectively, to reduce the current density, and
- 2) the inclusion of a sealed foil bag over the cell stack to prevent solvent vapors from interfering with the curing of the potting compound.

Cells of this final design were tested at room temperature and -30°C . The load currents were adjusted to maintain 5 watts during the 6 second pulse and a 15 milliwatts continuous drain. The cell voltages were monitored and the current adjusted daily to maintain this constant power load.

3.2 36 Volt Batteries

The 36 volt battery was designed to use the same electrodes and separators as the 35AH cells. The cells contained 3 cathodes and 4 anodes of the type shown in Fig. 3. The electrodes were stacked in same jig as the 35AH cells in Fig. 5. The cell assembly was then placed in the heat sealed foil laminate bag assembly shown in Fig. 17 and the electrode tabs welded to the cell terminal strips. After the electrolyte had been added to the cells the bags were heat sealed. The foil bags were then folded to resemble shallow cups as shown in Fig. 18. Leak tests were performed on each cell prior to stacking into the battery. Eleven cells were series connected by welding the terminal tabs and stacked (Fig. 19). The cell stack was placed in a plastic bag, leak tested, and then placed in the metal can. The cover terminals were welded to the battery tabs, the cover placed on the can, and the assembly was then potted in place.

Initial cells and the first complete batteries of this type were tested at room temperature and -30°C as indicated in 1.3.

A revised test schedule for these batteries was initiated. This test schedule is as follows:

- a) A continuous load of 15,000 ohm
- b) A 75 millisecond pulse on 57 ohm repeated each 100 seconds for 23 hours
- c) Every 24th hour: three 75 millisecond pulses on 57 ohm at 10 second intervals and one 15 second pulse on 114 ohm repeated each minute, (Fig. 14).

Special test equipment was constructed to perform this test at room temperature and -30°C , see Fig. 12 and the batteries are still discharging. In two battery tests conducted at USAECOM at -30°C , ten days of operation was obtained until the 57 ohm pulses fell below 20 volts. This corresponded to 1.0 AHr, 28 Whr and 20 watt hours per pound.

4.0 EXPERIMENTAL RESULTS

The 35AH cells constructed as described in section 3.0 were tested after various storage periods by discharging at constant power pulses of 5 watts in addition to a 15 milliwatt continuous load. The single cathode cells were similarly tested at densities corresponding to the pulse and continuous loads for the 35AH cells. The constant power discharges of the 35AH cells were carried out by adjusting the current to the values given in Tables 1 and 2 Appendix III on a daily basis.

Since the 35AH cells were constructed using 13 cathodes in the first prototype and 20 cathodes in the final design, two sets of current values were used for testing single cathode cells as given in Tables 3, 4, 5, and 6, Appendix III. These current values are $1/13$ and $1/20$ of the 35AH cell currents, respectively. All discharge curves for cells and batteries and the test summaries are given in Appendix III.

The discharge schedule specified for the 35AH cells was 15 milliwatt continuous and a 6 second duration 5 watt pulse every 10 minutes (1% pulse duty cycle). In the later stages of the project this discharge schedule was modified to a 6 second duration 5 watt pulse each minute (10% duty cycle). This change was made to provide the cell performance data required for evaluations in a much shorter time. Comparison tests indicated that the cell performance did not diminish by applying this more severe test.

The specified test schedule for the 36 volt battery is as follows: continuous load of 15,000 ohms and a 65 millisecond pulse load of 57 ohms repeated 1200 times per day. The equipment for performing this test is given in Fig. 12.

The evaluation tests were performed on the 36 volt battery using this test. The tests on 3 cathode cells for this battery were performed using the equivalent discharge conditions of 1000 ohms continuous and 4 ohms pulse load for each cell on the same schedule. When the first batteries

were delivered the test schedule was revised as indicated in 3.2 and detailed in Fig. 14 in which the pulse duration was increased to 75 milliseconds and 15 second pulse loads on 114 ohms were added.

The required service from the 36 volt batteries on the initial test schedule was 38 days or 912 hours. On the revised test schedule 20 days or 480 hours is required. Room temperature tests only were specified, but since -30°C operation was anticipated low temperature tests were also performed. (Present USAECOM battery specifications require a minimum of 20 days service at any temperature from -30°C to 50°C .)

- 4.1 Circular Cell Tests (1 cathode, 2 anodes). Cells were made containing cathode B and electrolyte H (batch 700723). The cathodes used in these cells were prepared using a controlled particle size powder for molding to give a uniform die cavity fill. The testing of these cells was intended to evaluate the performance of cathodes made by this process.

The fresh test discharge curves at room temperature and -30°C are given in Figs. 20 and 21. The energy density for these cells extrapolated to the 35AH cell size was 19.6 whr/lb at room temperature and 43.5 whr/lb at -30°C . The cell which performed poorly at room temperature was examined and the cathode was found to be cracked, and it had broken away from the current collector. This condition was not found when the -30°C test cell was examined. The cells from this batch that were intended for tests after 55°C storage were destroyed when a temperature controller failed allowing the 55°C storage chamber to reach 150°C .

Cells containing cathode B and electrolyte J (batch 700807) were assembled and tested. These cells were made to elaborate on the preliminary results obtained with this type of electrolyte in rectangular cells (batch 700309), see Q-3. The fresh test discharge curves for these cells at room temperature and -30°C are given in Figs. 22 and 23. The extrapolated energy densities for these cells were 97.4 whr/lb at room temperature and 40.8 whr/lb at -30°C . The cells from this batch that were stored at 55°C were also destroyed when the temperature controller failed.

Cells were made containing cathode B and electrolyte K (batch 700811). These cells were made to elaborate on the preliminary results obtained with

this type of electrolyte in rectangular cells (batch 700309), see Q-3. The fresh -30°C test constant power (see Tables 3 and 4) discharge curve is shown in Fig. 24. The first discharge pulse on these cells resulted in a cell voltage below the 1.5 volt cutoff. Subsequent pulses were above the 1.5 volts. The extrapolated energy density for this test was 42.8 whr/lb at -30°C .

The discharge of cells from this batch that were stored for 3 months at room temperature and then discharged at -30°C is indicated by Fig. 25. This discharge was carried out at constant power using a current density corresponding to the 20 cathode cell. The extrapolated energy density for this test was 65 whr/lb at -30°C .

The cells from this batch discharged at -30°C after 3 months at 45°C had an initial pulse where the cell voltage was below the 1.5 volt cutoff. The discharge curve for this test is given in Fig. 26. This test was also carried out at constant power at current densities corresponding to the 20 cathode cell. The extrapolated energy density for these cells was 40 whr/lb at -30°C . This test was performed to determine the comparative effect of 45°C and 55°C storage. This test was not performed on cells containing other electrolyte types due to schedule limitations.

The cells from this batch that were stored for 3 months at 55°C before discharge at -30°C remained below the 1.5 volt cutoff. These tests were discontinued. Further use of type K electrolyte was not indicated by the poor performance of these cells after 55°C storage.

Cells from various batches were examined after storage and discharge in an effort to determine what construction improvements could be made to improve the cell performance. This examination revealed that some of the cathodes had blistered and lost contact with the collectors. Cells made from improved cathodes (batch 701010) were discharged at room temperature and -30°C . The discharge curves are given in Figs. 27 and 28. The extrapolated energy densities for these cells were 96 whr/lb at room temperature and 54 whr/lb at -30°C .

- 4.2 35AH Cells - 13 Cathodes (LB-35). Cells were made using 13 cathodes and 14 anodes. These electrodes were assembled and sealed in cans as indicated in Fig. 16.

The cells assembled using 13 type B cathodes and H electrolyte were pulse discharged at room temperature (batch 700529). The test currents were 5 ma continuous and 1.9 ampere pulse. The discharge curve for this cell is given in Fig. 29. The energy density for this cell was determined by using the max weight allowed since the cell was assembled without potting compound. The energy density determined from this weight and the discharge curve was 27 watt hours per pound.

A similar cell from batch 700601 was discharged at -30°C using the same currents for continuous and pulse drains. The discharge curve is given in Fig. 30. The premature and sudden failure of this cell was found to be a result of a broken tab on the cathode terminal. Subsequent discharges of cathodes removed from this cell indicated a considerable residual capacity.

Cells were assembled using cathode B and electrolyte H (batch 700618). These cells were discharged fresh at a constant power as determined from the cell voltage and current tables, Tables 1 and 2 (Appendix III). The discharge curves are given in Figs. 31 and 32. The energy density for the room temperature cell test was 50 watt hours per pound. The energy density for the -30°C cell test was 40 watt hours per pound. Both energy densities are based on the maximum cell weight of 0.65 lb. The actual cell weights were 0.57 lb. No potting compound was used in the assembly of these cells.

The losses in energy density relative to the expected values led to a re-examination of the design to determine all possible IR losses. The resistance of the electrode tab material was measured and it was found to contribute losses of 0.2 to 0.3 volts at the cutoff voltage. These tabs were changed to reduce the resistance in subsequent cells.

Cells were assembled containing cathode B and electrolyte H (batch 700806). These cells were tested at room temperature and -30°C . The constant power discharges for these cells are indicated in Figs. 33 and 34. The energy densities for these cells were 55 whr/lb at room temperature and 27 whr/lb at -30°C .

Cells were made containing type B cathodes and H electrolyte (batch 700821). These cells also had the improved tabs between the electrodes and the cell terminals. The constant power discharge curves of these cells are given in Figs. 35 and 36. The energy densities for these cells were

60 whr/lb at room temperature and 33 whr/lb at -30°C . The pulse test results for 13 cathode 35AH cells are summarized in Table 8.

- 4.3 35AH Cells - 20 Cathodes (LB-35). A cell was made containing 20 type B cathodes and electrolyte H (batch 700923). This cell also had improved low resistance tabs connecting the electrodes to the cell terminals. The constant power discharge performance of this cell on 10% duty cycle is given in Fig. 37. The energy density for this cell was 89 whr/lb.

The cathodes used in this cell were the same as used in the 13 cathode cells and therefore nearly filled the cell can. Canning this cell was very difficult due to the larger electrode stack. Leakage of the electrolyte was noted after the discharge of this cell. Examinations indicated that the inner foil bag had been punctured during the canning operation. Subsequently, 20 cathode cells were made with thinner cathodes, to reduce the canning problems.

Cells were made using 20 type B cathodes and electrolyte R (batch 700928). These cells were discharged at constant power as indicated in Figs. 38, 39, 40 and 41. The fresh discharge energy densities for these cells were 84.5 whr/lb at room temperature and 22 whr/lb at -30°C . The energy densities for discharges after 1 week of storage at 55°C were 57 whr/lb at room temperature and 13 whr/lb at -30°C .

Examination of the cells after discharge revealed the blistered and cracked cathodes noted in paragraph 4. 1.

Cells were made containing 20 type B cathodes and electrolyte R (batch 701008). A representative constant power discharge at room temperature is given in Fig. 42. The energy density for this cell was 72.5 whr/lb.

Cells with improved cathodes of type B and electrolyte R (batch 701012) were constant power discharged fresh at room temperature; a representative discharge curve is given in Fig. 43. The energy density for this cell was 102 whr/lb.

Cells from batch 700928 were subjected to vibration tests as specified in Q-1. During this test the open circuit voltage was monitored and no change was noted. After the vibration test the cells were returned to our laboratory (approximately 3 weeks had elapsed since the cells were constructed) and constant power discharged at room temperature. A

representative discharge curve is given in Fig. 44. The energy density of this cell was 80 whr/lb.

A summary of the 20 cathode 35AH cell tests is given in Table 9 (Appendix III).

- 4.4 Circular Cells - 36 Volt Battery. As indicated in paragraph 1.3 and 3.2 the objectives of the contract were changed to include 15 batteries. Cells for these batteries were assembled and tests began prior to the receipt of the battery cases and covers. The discharge of these cells is being carried out at room temperature using 1000 ohm continuous load and 4 ohm load pulsed for 65 milliseconds, 1200 times per day. The discharge equipment for this test is given in Fig. 12.

The cells were assembled using three type B cathodes with 4 anodes and electrolyte R (batch 701203). These cells were discharged at room temperature using the test already described. The discharge curve, Fig. 45, indicates the continuing cell test in which the voltage is about 2.0 volts after 161 days + 16 hours (3880 hours). The cells are still discharging. The service required for the battery is 912 hours to a 20 volt cutoff. The corresponding cell cutoff voltage in an eleven cell battery is 1.82 volts.

Cells containing 3 type B cathodes and electrolyte R (batch 701222) were made in foil laminate containers shaped to fit the battery case. These cells were discharged on the same schedule and loads. The discharge curve for this continuing test is given in Fig. 46.

- 4.5 36 Volt Batteries (LB-5-36). A 36 volt battery was assembled from cells from batch 701222 (see above). This battery was discharged on the same schedule using a 15,000 ohm continuous load and a 56 ohm pulse load. The discharge curve for this battery test is given in Fig. 47 and the discharge has gone 97 days + 22 hours (2350 hours) and is still running. This battery weighs 1.43 pounds including case, cover and potting compound.

Cells were assembled containing 3 type B (batch 710223) cathodes and electrolyte R. These cells were used to assemble the final ten batteries. Cells from this batch were tested at -30°C using the revised test schedule described in paragraph 3.2 and detailed in Fig. 14. A representative discharge curve is given in Fig. 48. The requirement service for this battery is 480 hours at room temperature on this revised test schedule. These

cells were discharged for 24 days + 16 hours (592 hours) at -30°C to a 1.8 volt cutoff during the 15 second pulse on 8 ohms.

The voltage on the 75 msec pulse at 4.0 ohms was checked periodically during the test and was found to be higher in all cases than the voltage on the 15 second pulse. The discharge curve of Fig. 48 gives the continuous load (1000 ohms) voltage and the 15 second (80 ohms) pulse voltage.

5.0 CONCLUSIONS

5.1 35AH Cells (LB-35).

- 1) Cells can be made that will deliver 35AH and 95 watt hours per pound at room temperature.
- 2) These cells will also deliver 55 watt hours per pound when discharged at -30°C .
- 3) Reduced temperature storage is recommended for these cells.
- 4) Seventy of these cells were delivered for evaluation.

5.2 36 Volt Batteries (LB-5-36).

- 1) A 36 volt battery can be made from the LB-35 cell components that exceeds the performance specified requirements of 912 hours of service.
- 2) Fifteen of these batteries have been delivered for evaluation.

5.3 Projected Service - 36 Volt Batteries.

The test results on improved cells and the discharge tests which are continuing indicate that 36 volt batteries tested on the revised test schedule will give the following service to the 20 volt cutoff: (480 hours required)

- | | |
|--------------------------|-------------------|
| 1. Room temperature | 900 to 1000 hours |
| 2. -30°C | 550 to 600 hours |

A maximum storage temperature for these cells is 45°C or 113°F .

6.0 MANPOWER EXPENDED

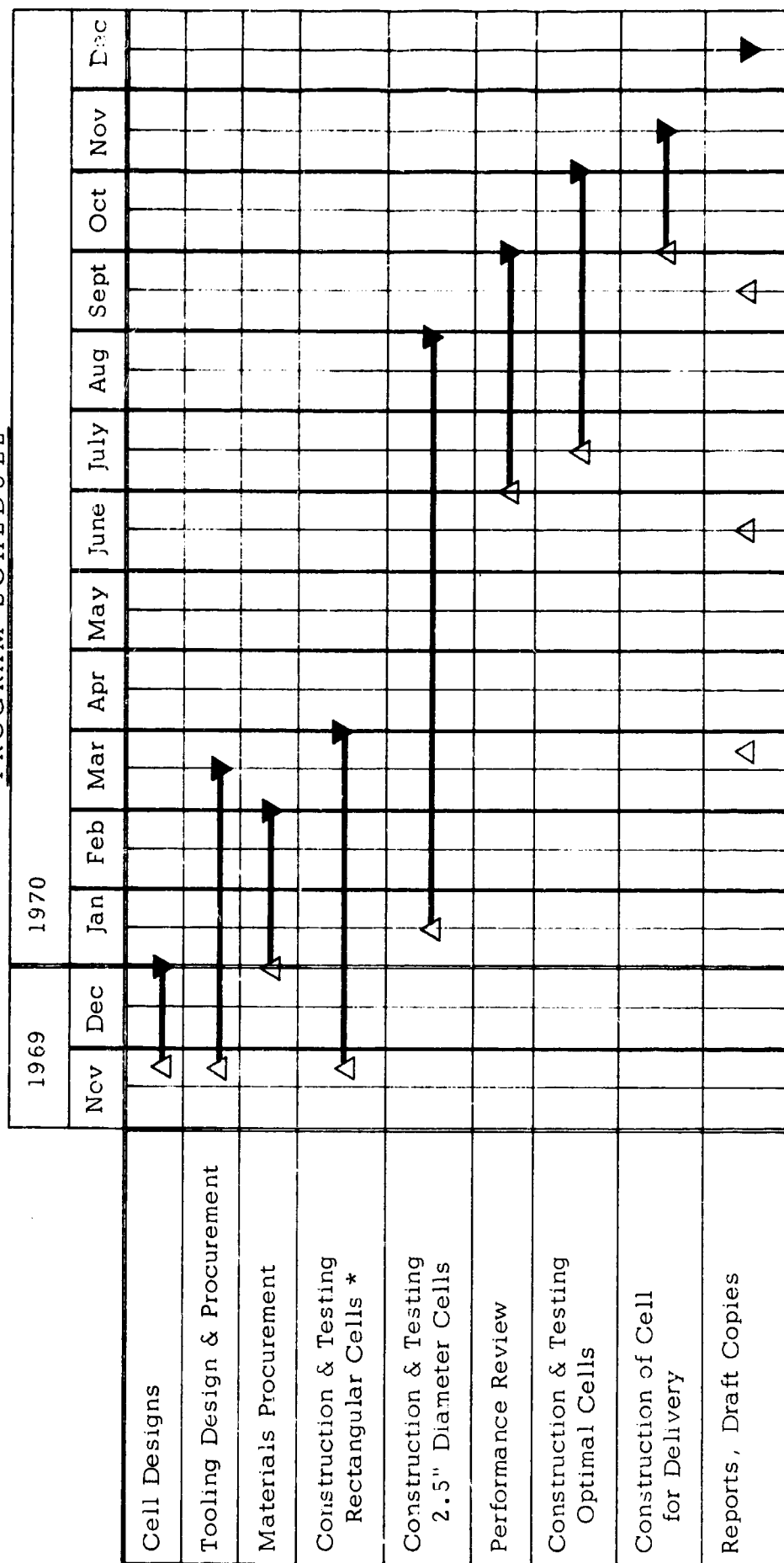
Group Manager Electrochemistry	P. Bro	240 hours
Principal Investigator	A. N. Dey	480 hours
Project Director	L. F. Athearn	1544 hours
Technician	R. Bessett	1992 hours
Technician	J. Him	1376 hours
Technician	D. Veinot	4 hours
Machinist	E. Klosterman	48 hours
Machinist	R. Saladino	8 hours

SUPPLEMENT

1. Conferences

- a) Conference at Laboratory for Physical Science, P. R. Mallory & Co. Inc., Burlington, Mass. on September 18, 1970 with Project Monitor to discuss cell performance and progress.
- b) Conference at Ft. Monmouth on November 4, 1970 with Project Monitor, representatives of project management group and representatives of DCPG.
- c) Conference at Ft. Monmouth on November 23, 1970 with Project Monitor at which time 70 cells were delivered.
- d) Conference at Ft. Monmouth on February 5, 1970 with Project Monitor to discuss cell performance and deliver 5 batteries.
- e) Conference at Ft. Monmouth on March 9, 1970 with Project Monitor to discuss cell and battery performance and deliver 10 batteries.

PROGRAM SCHEDULE



* Evaluation of the optimum proprietary system and optimum proprietary construction techniques to meet the particular electrochemical requirements of subject contract.

FIG. 1

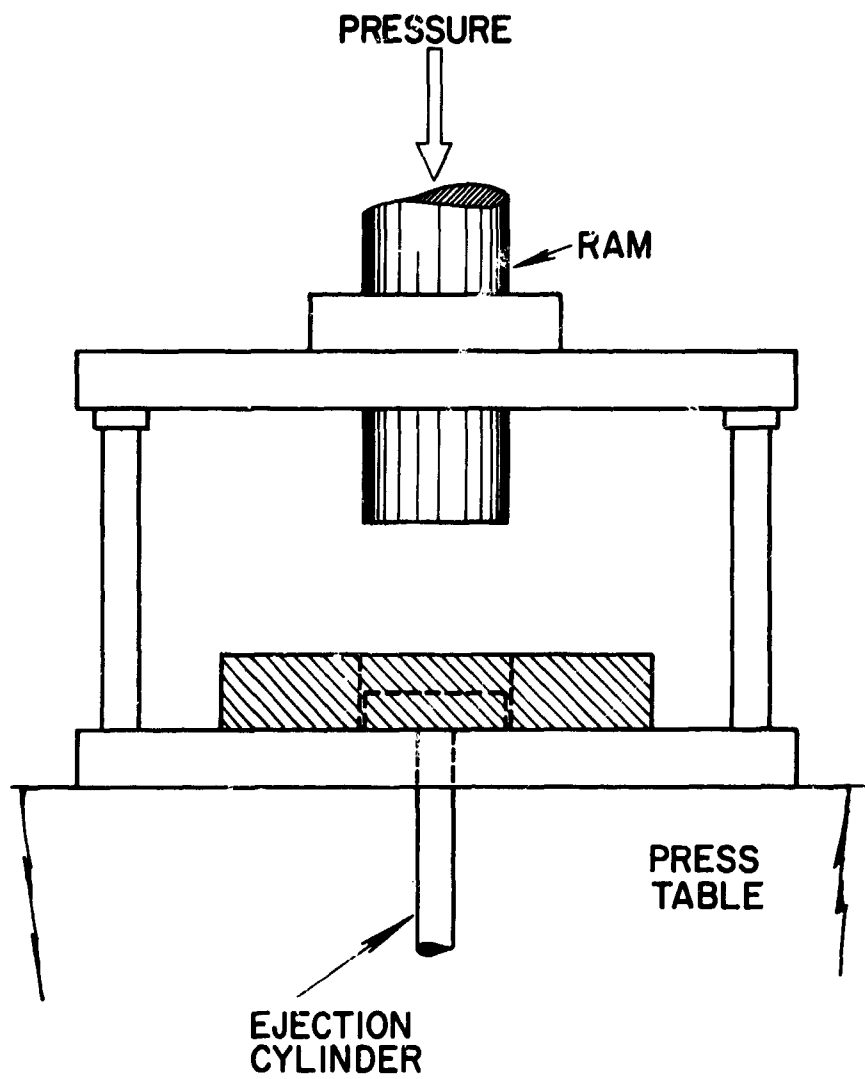


FIG. 2 CATHODE MOLDING DIES

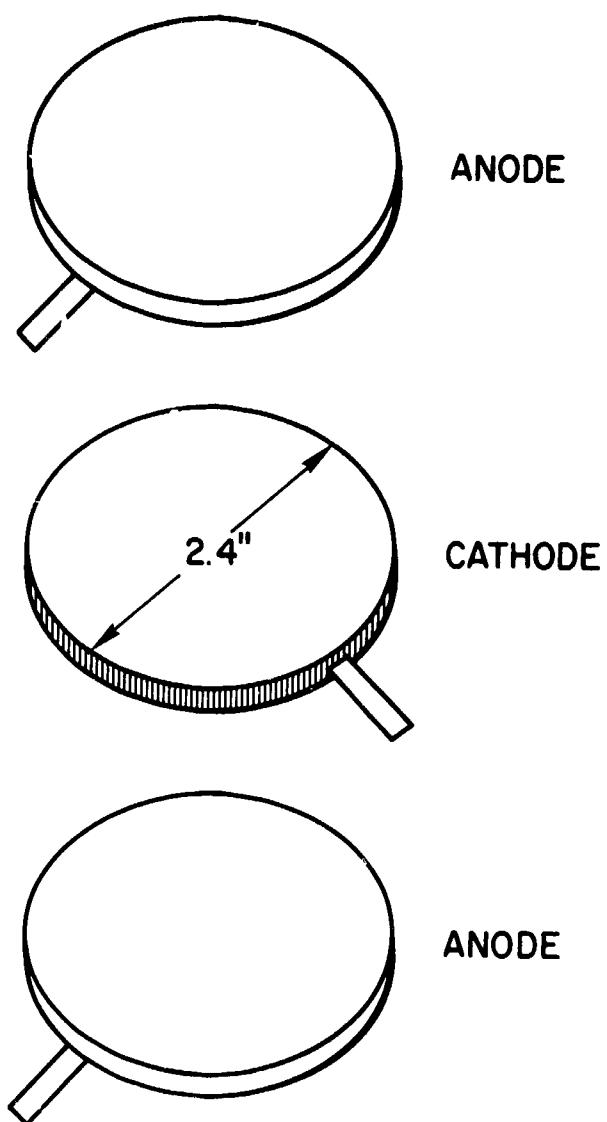


FIG. 3 ANODE AND CATHODE

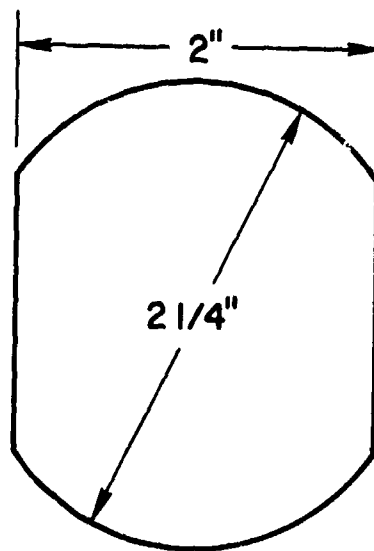


FIG. 4 ANODE COLLECTOR GRID

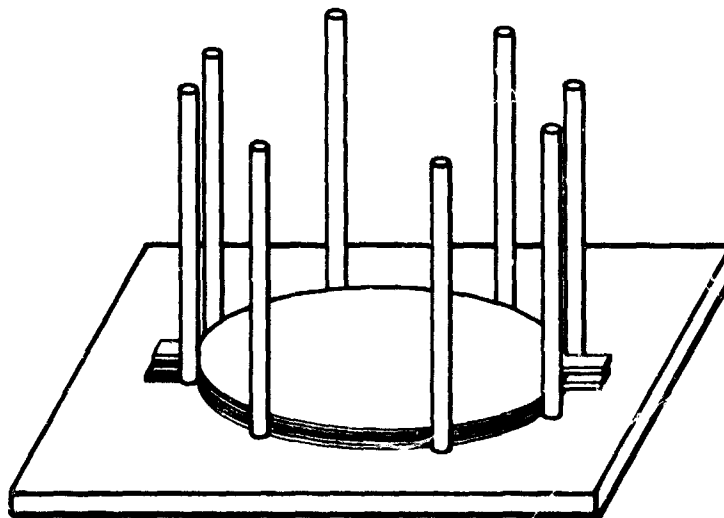
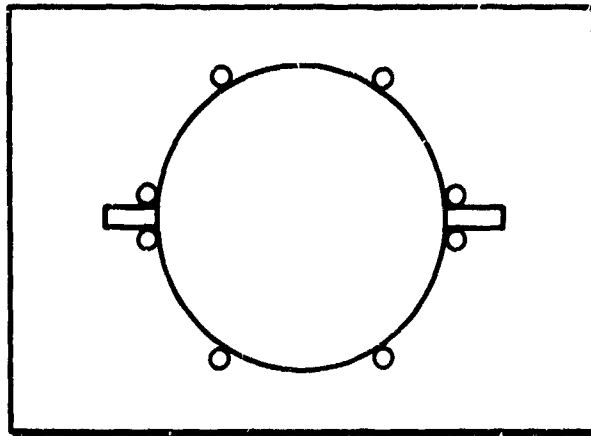
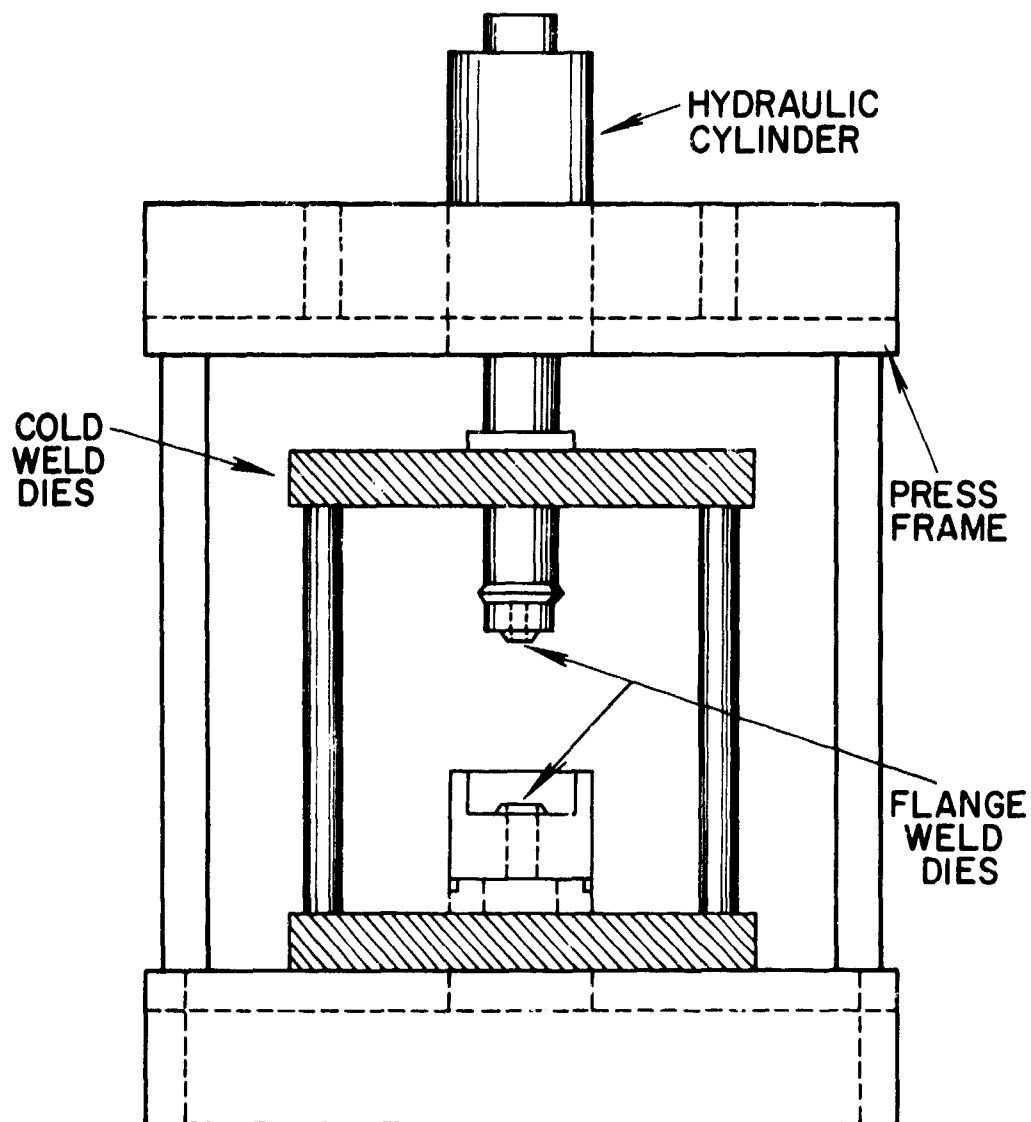


FIG. 5 STACKING JIG



FLANGE WELD TOOLS

FIG. 6

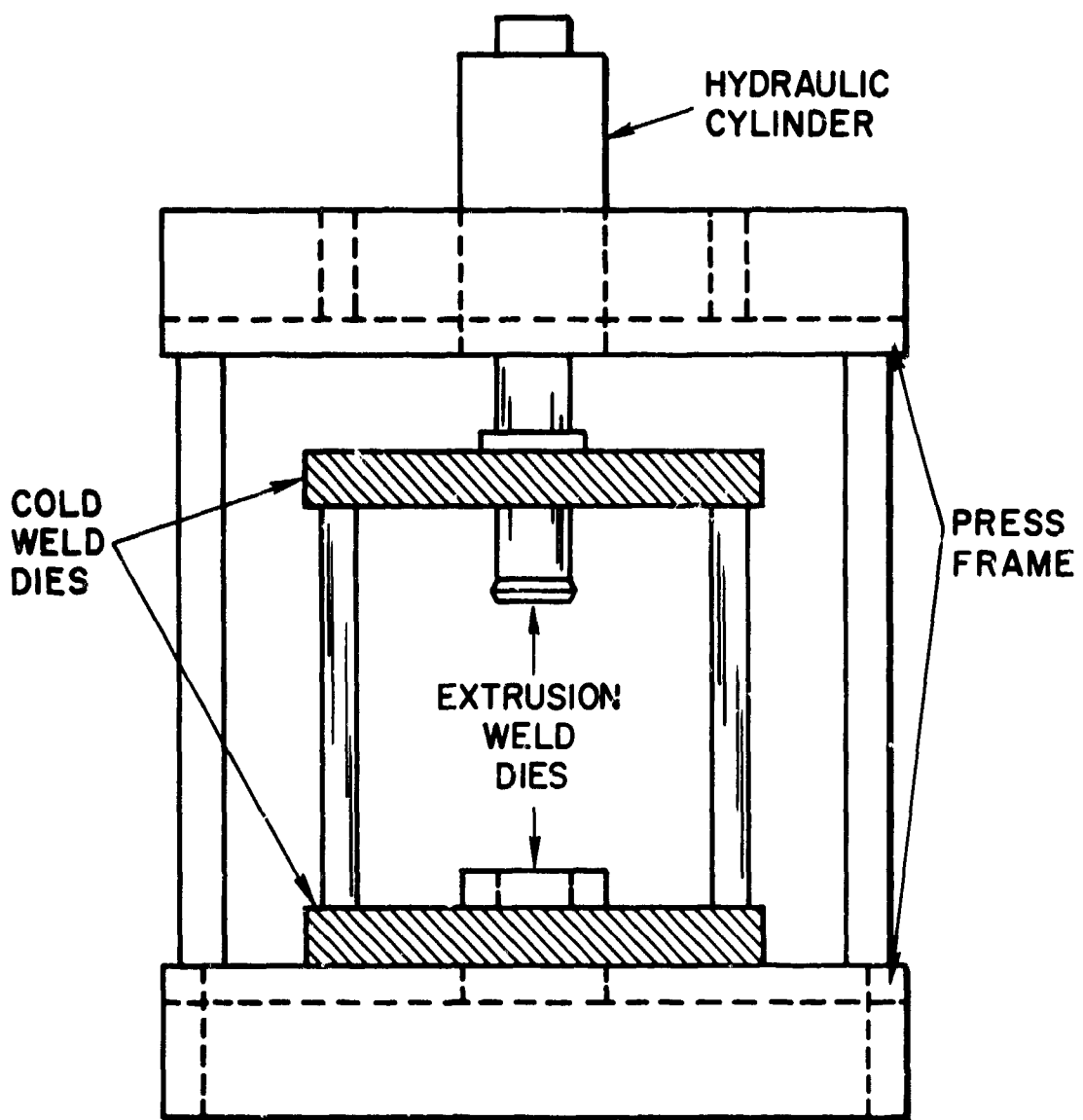
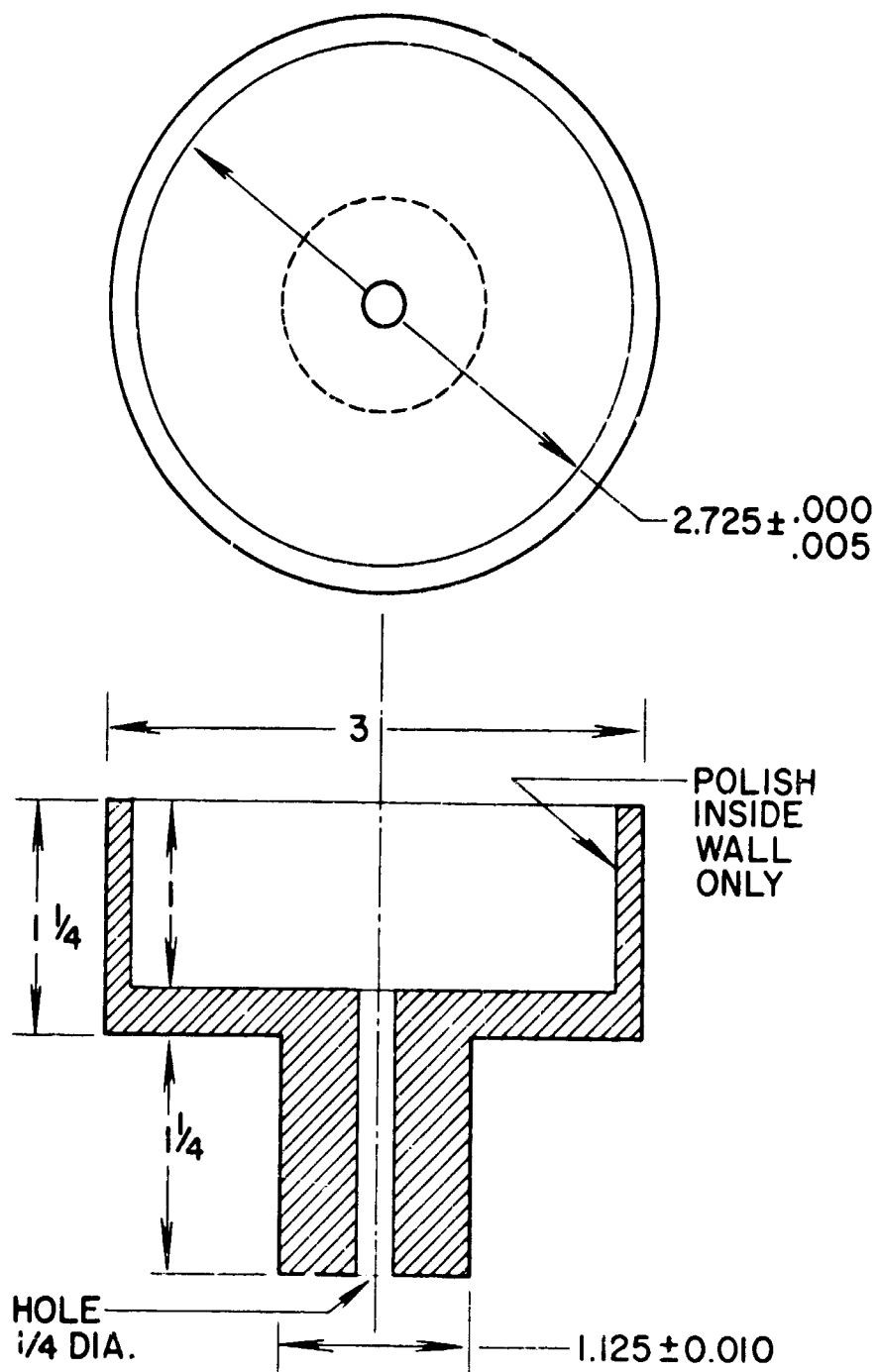
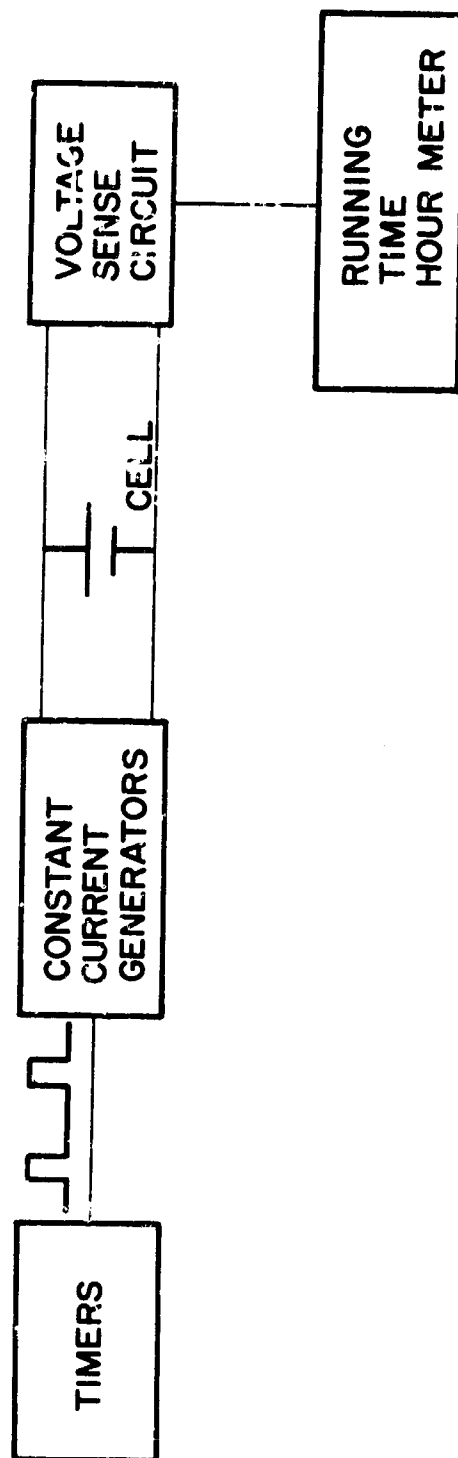


FIG. 7 EXTRUSION WELD TOOLS



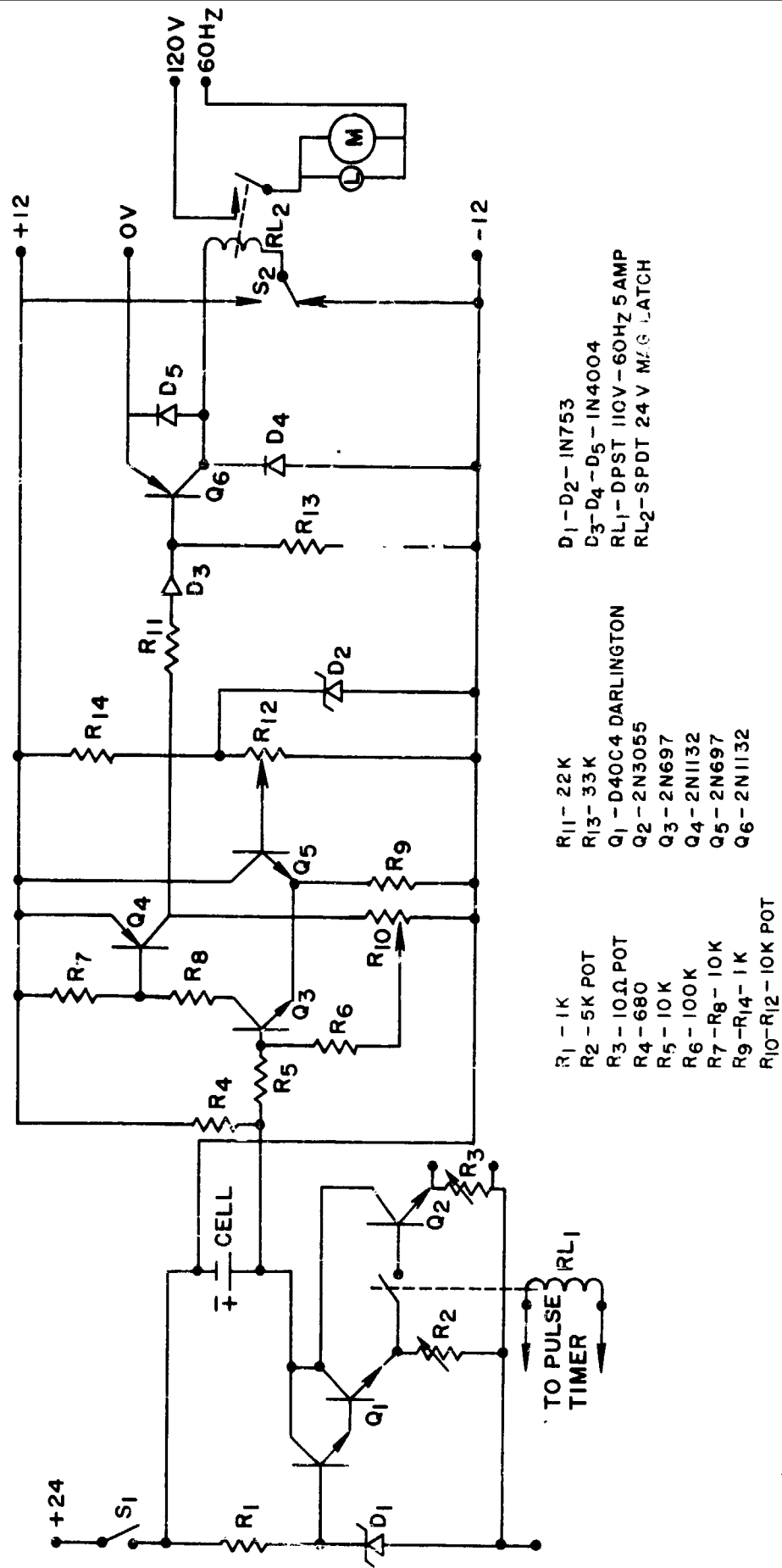
LEAK TEST ADAPTER

FIG. 8



CELL TEST EQUIPMENT BLOCK DIAGRAM

FIG. 9



- | | | |
|---------------------------------------------|-----------------------------------|-----------------------------------------------------------|
| R ₁ - 1K | R ₁₁ - 22K | D ₁ - D ₂ - IN753 |
| R ₂ - 5K POT | R ₁₃ - 33K | D ₃ - D ₄ - D ₅ - IN4004 |
| R ₃ - 10Ω POT | Q ₁ - D40C4 DARLINGTON | RL ₁ - DPST 110V - 60HZ 5AMP |
| R ₄ - 680 | Q ₂ - 2N3055 | RL ₂ - SPDT 24V MFG LATCH |
| R ₅ - 10K | Q ₃ - 2N697 | |
| R ₆ - 100K | Q ₄ - 2N1132 | |
| R ₇ - R ₈ - 10K | Q ₅ - 2N697 | |
| R ₉ - R ₁₄ - 1K | Q ₆ - 2N1132 | |
| R ₁₀ - R ₁₂ - 10K POT | | |

CELL TEST CIRCUIT

FIG 10

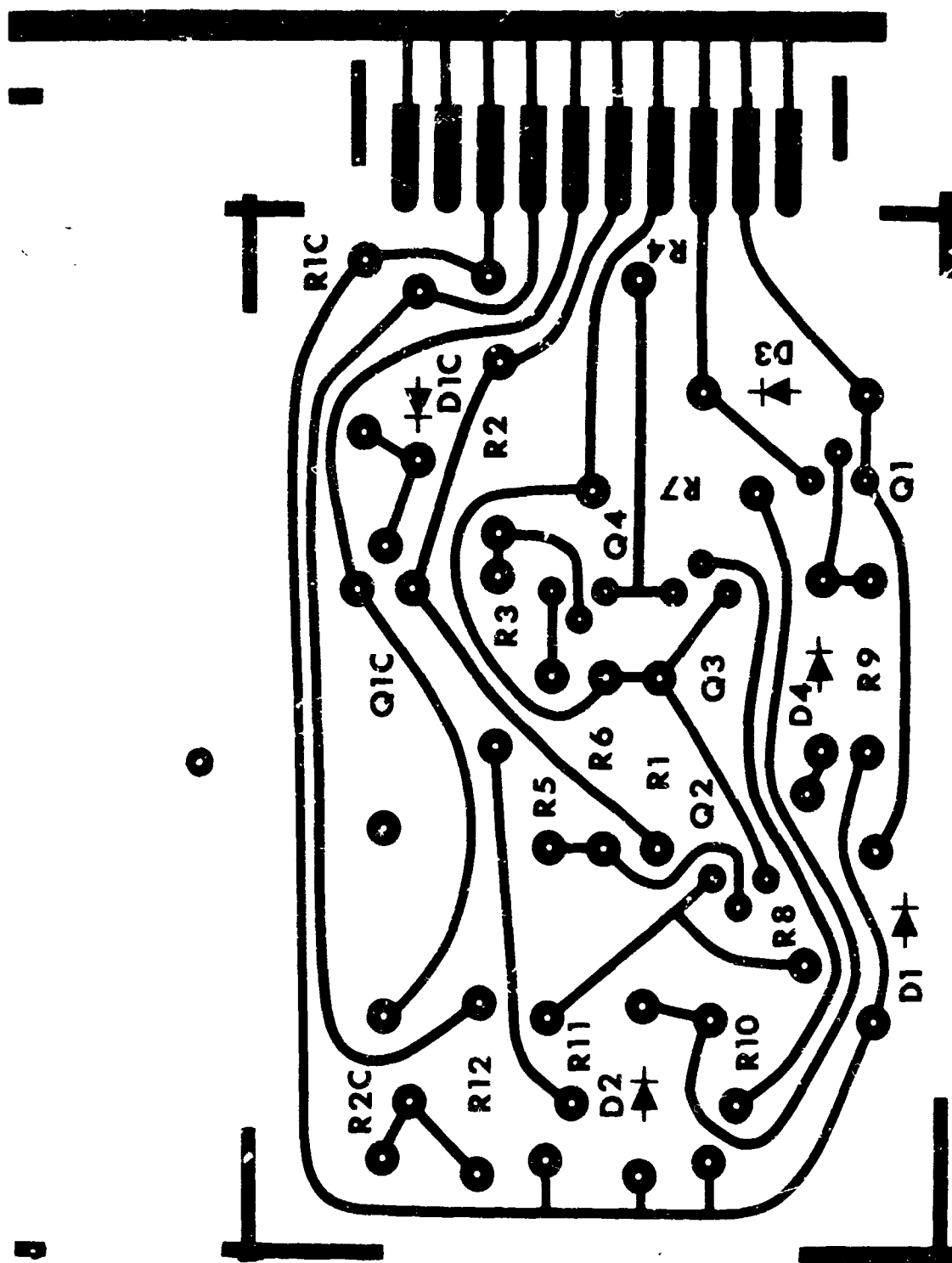
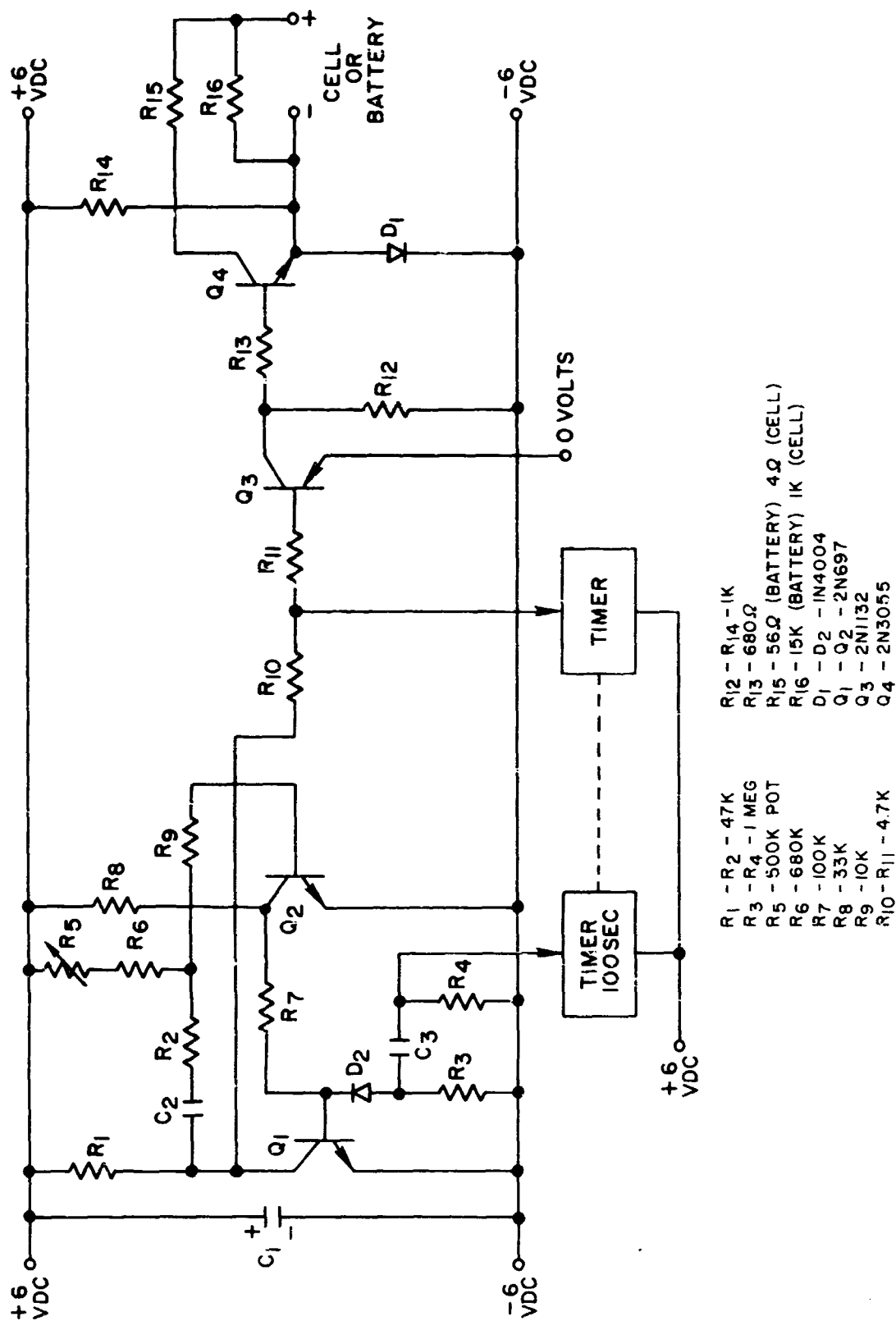
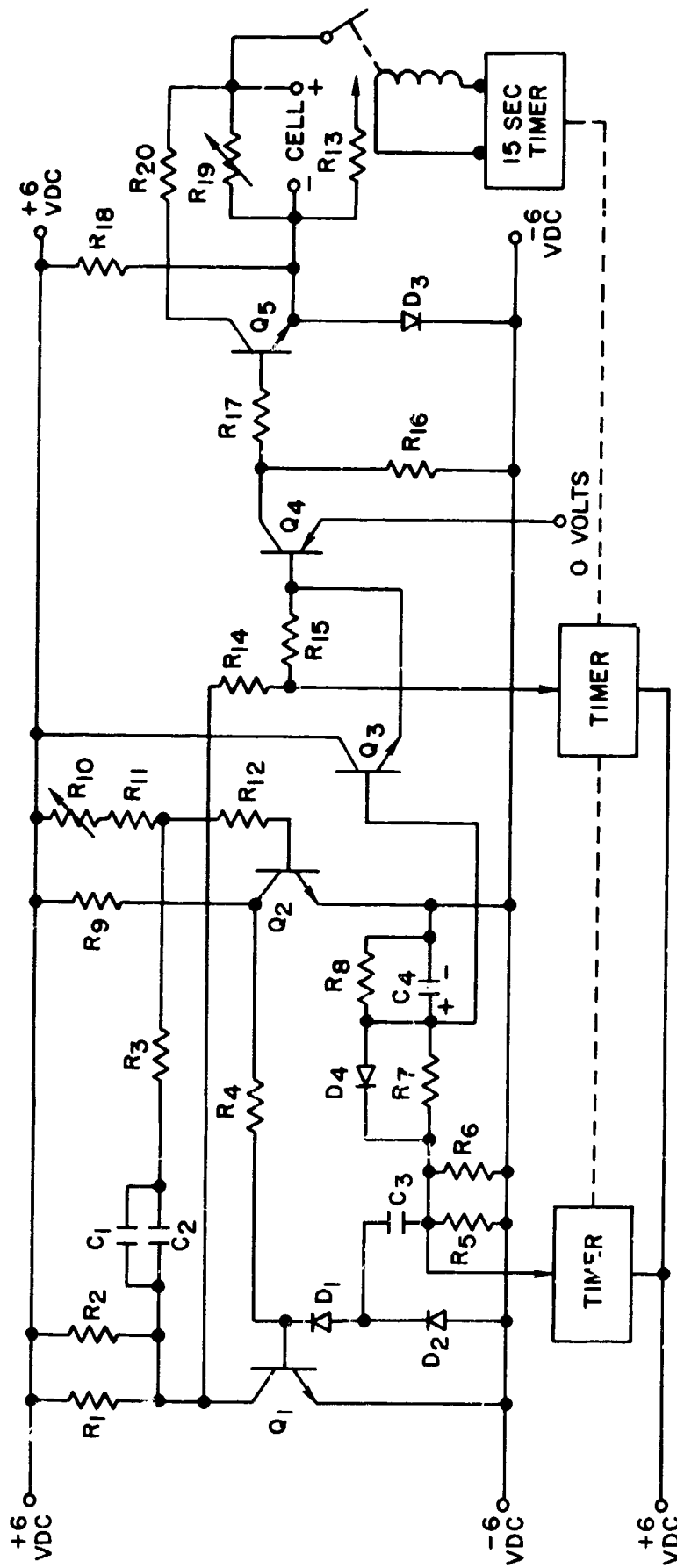


FIG. II CELL TEST PRINTED CIRCUIT BOARD

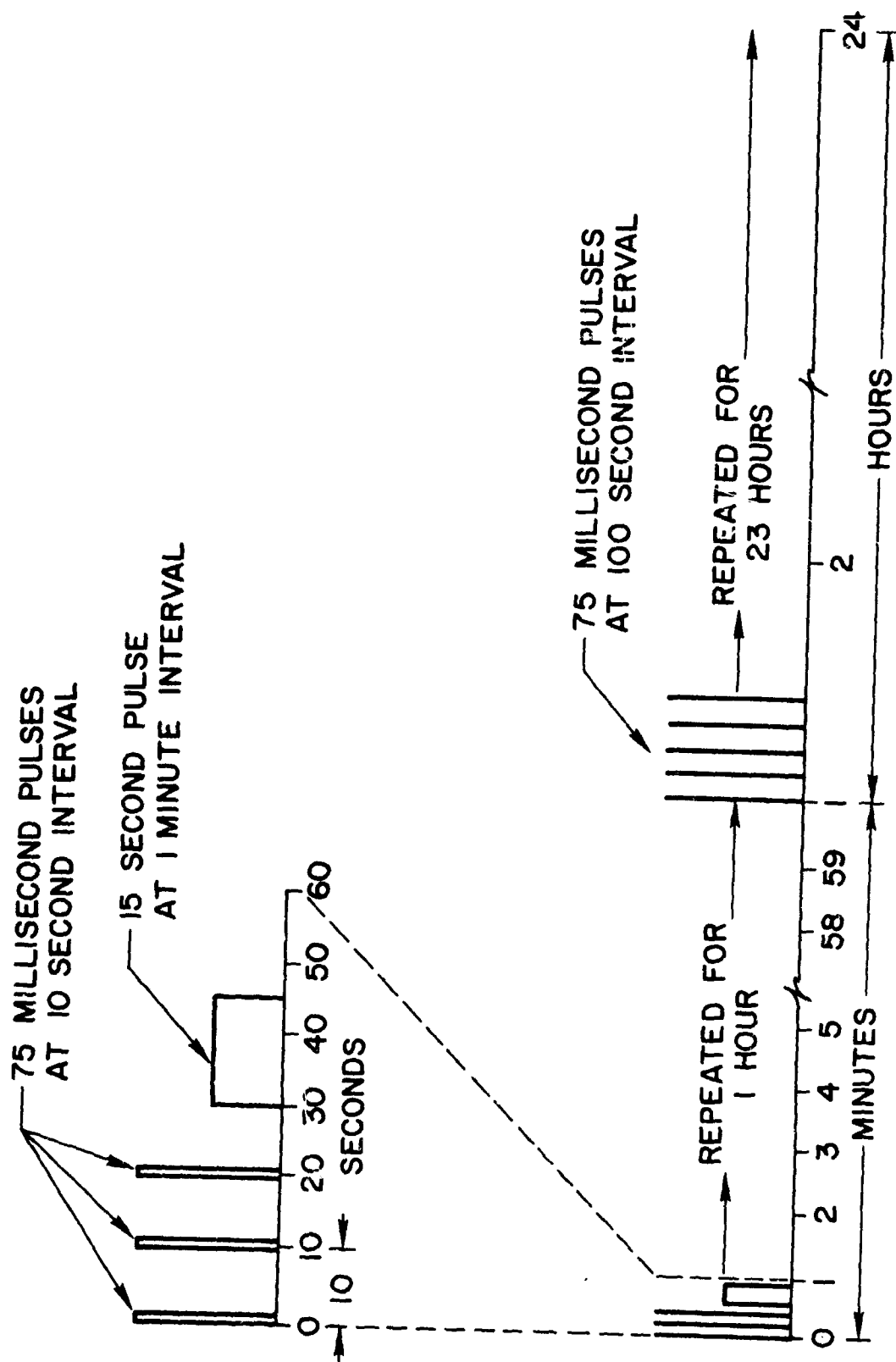


65 MILLISECOND PULSE TEST CIRCUIT
36 VOLT BATTERY
FIG 12



- | | | |
|----------------|--------------------------------|---------------|
| R1 - R12 - 10K | R13 - 114Ω (BATTERY) 8Ω (CELL) | Q5 - 2N3055 |
| R2 - R3 - 47K | R14 - R15 - 4.7K | C1 - 0.47 μfd |
| R4 - 100K | R16 - R18 - 1K | C2 - 0.1 μfd |
| R5 - 1 MEG | R17 - 680Ω | C3 - 1 μfd |
| R6 - R11 - 15K | R19 - 15K (BATTERY) 1K (CELL) | C4 - 25 μfd |
| R7 - 68K | R20 - 56Ω (BATTERY) 4Ω (CELL) | |
| R8 - 4.7 MEG | D1 - D2 - D3 - D4 - IN4004 | |
| R9 - 33K | Q1 - Q2 - Q3 - 2N697 | |
| R10 - 500K | Q4 - 2N1132 | |

REVISED TEST CIRCUIT
36 VOLT BATTERY
FIG 13



REVISED TEST SCHEDULE - 36 VOLT BATTERY
FIG. 14

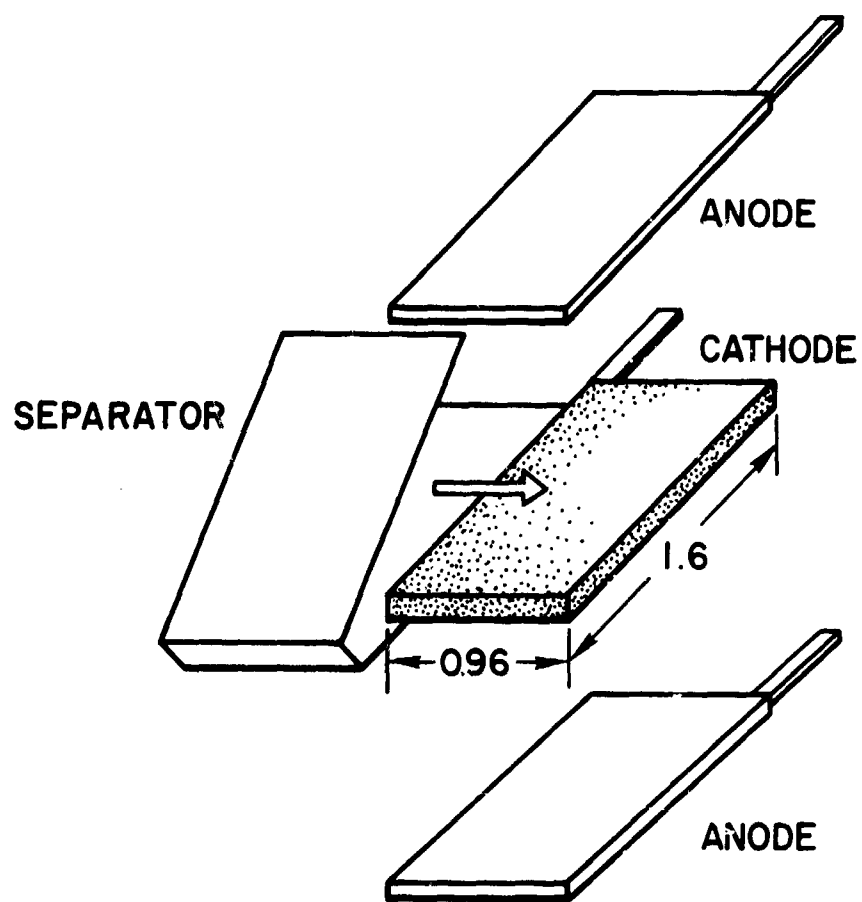


FIG. 15 RECTANGULAR CELL ASSEMBLY

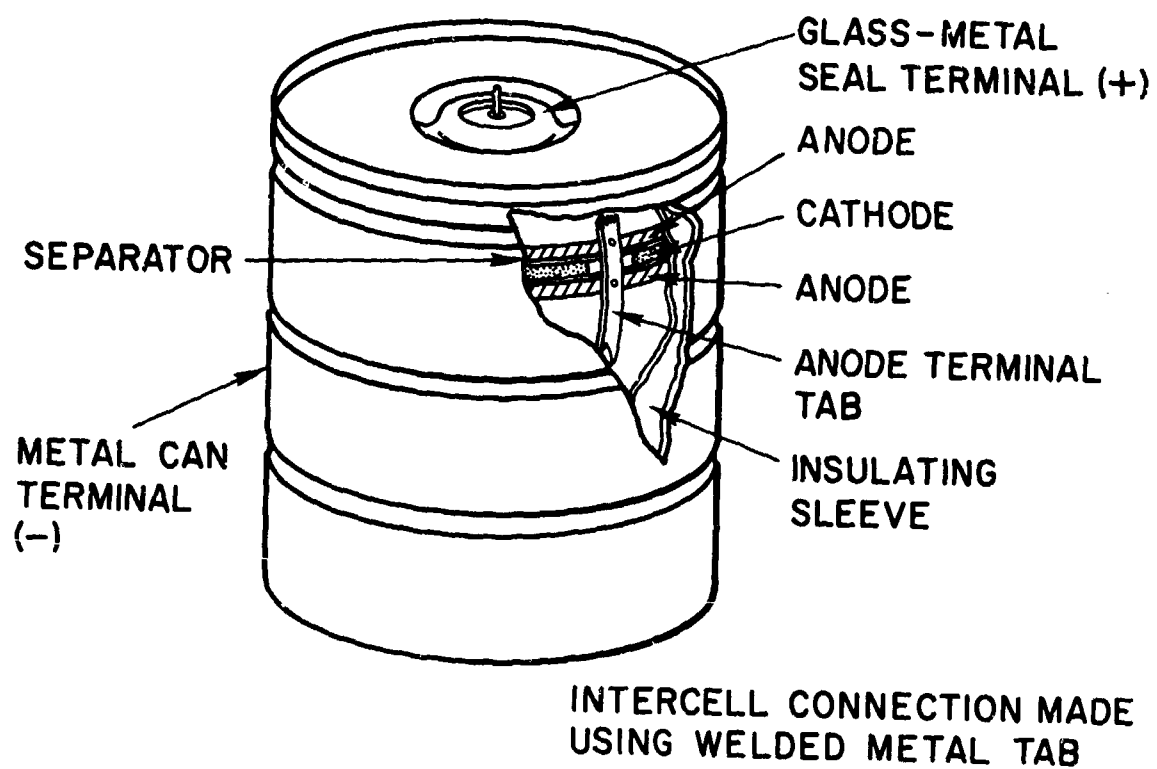


FIG. 16 CELL DESIGN

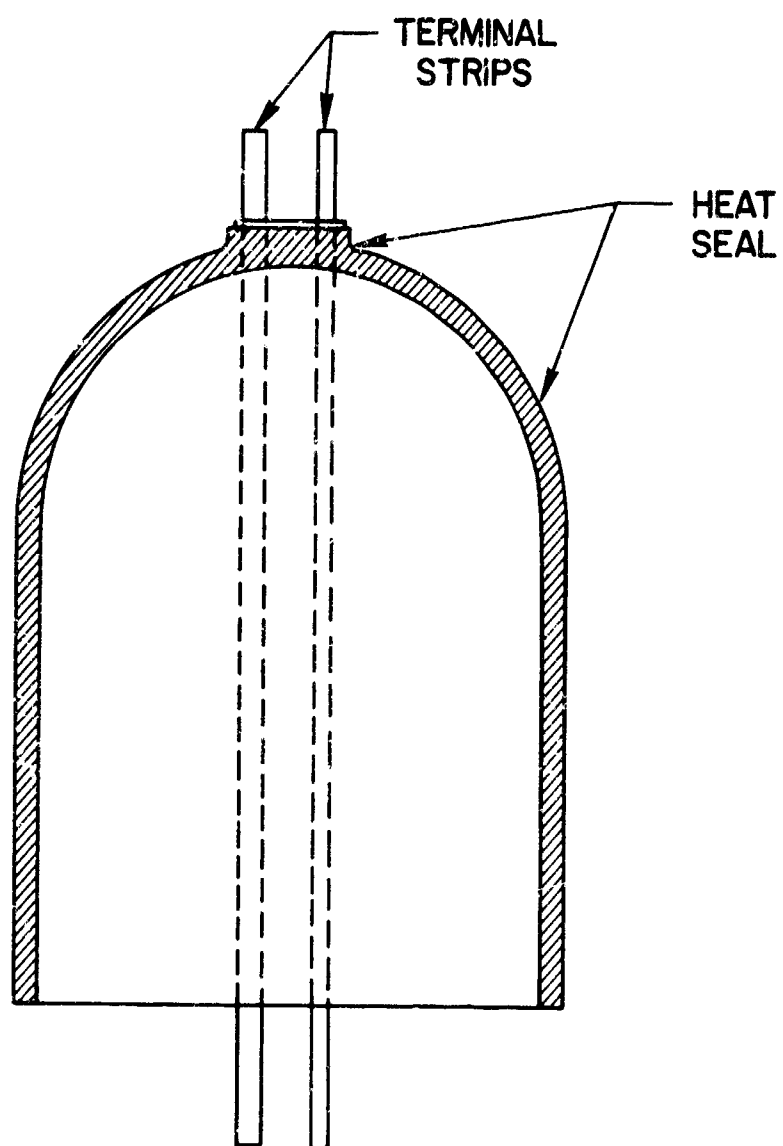


FIG. 17 FOIL LAMINATE CELL BAG

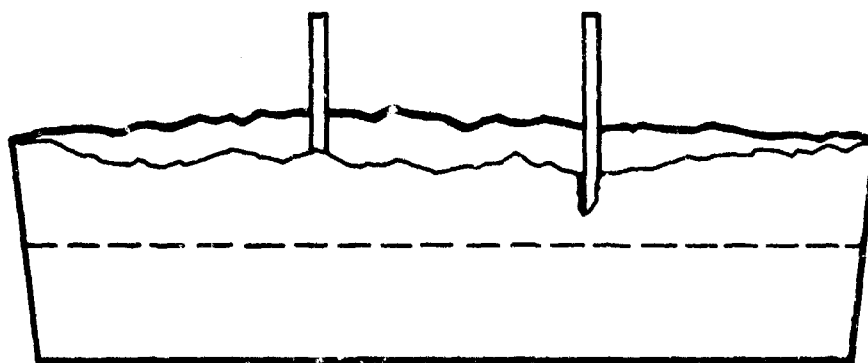


FIG. 18 FOIL LAMINATE CELL CUP

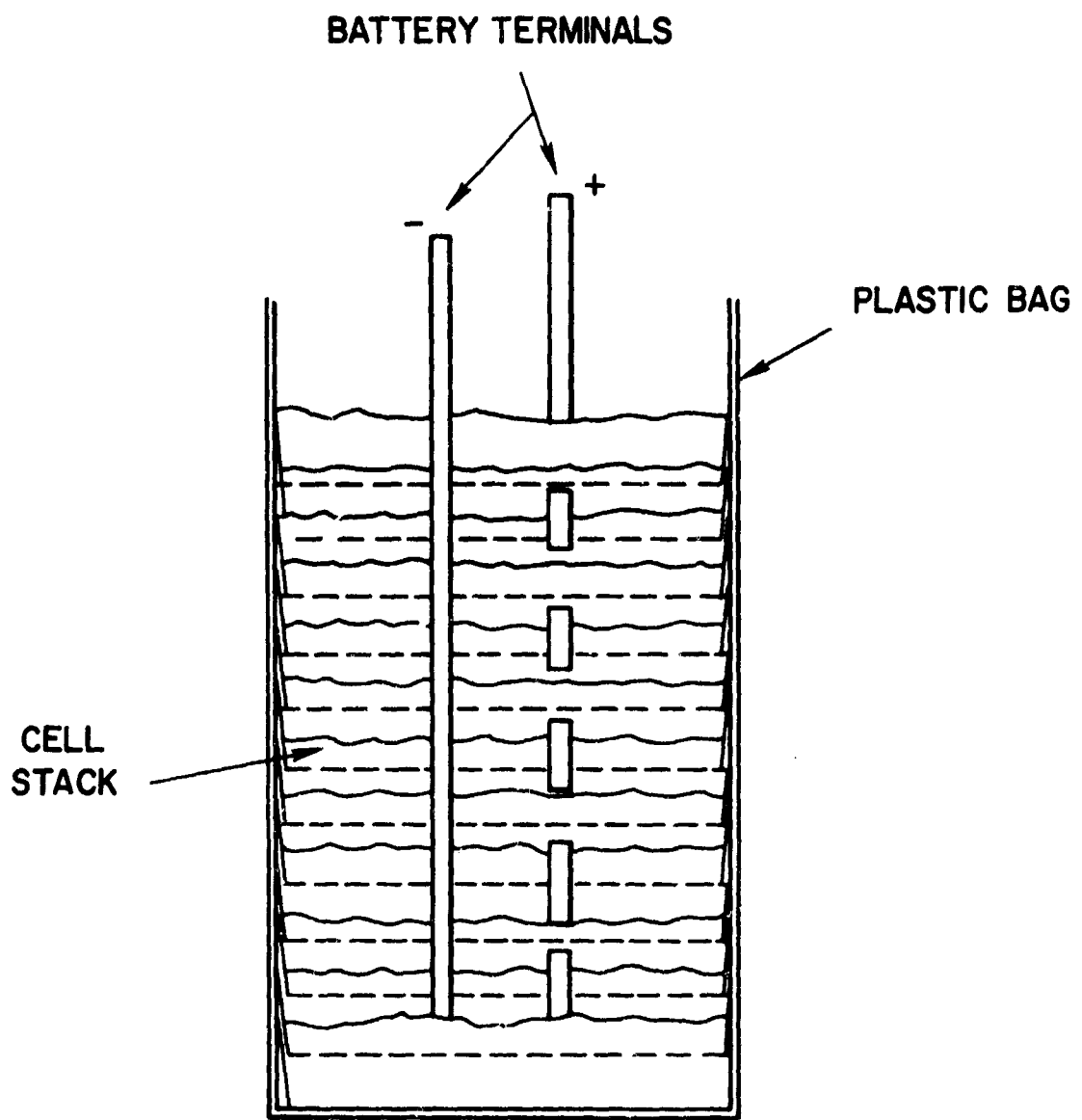


FIG. 19 36 VOLT BATTERY STACK

APPENDIX I

OBJECTIVES

The purpose of this project is to develop an organic electrolyte cell that will operate over the extended temperature range required for military applications and deliver 100 watt hours per pound. The specific requirements for this cell are:

- 1) Physical: Size 2.55" dia. x 2.5 high max. weight 0.65 pounds max.
- 2) Environmental: Discharge at two temperatures, -20°F (-30°C) $+75^{\circ}\text{F}$ (20°C) and storage at $+130^{\circ}\text{F}$ (55°C) for 90 days.
- 3) Electrical: Two power levels steady state and pulse 1% duty cycle as follows.
 - a. average power, steady state, 15 milliwatts
 - b. average power, pulse, 5 watts
 - c. pulsing duty, 1%
 - d. pulse duration, 6 seconds
 - e. pulse frequency, one pulse every 10 minutes
 - f. service, 1000 hours minimum
 - g. steady state energy, 15 watt hours (min.)
 - h. pulse energy, 50 watt hours (min.)
 - i. total energy, 65 watt hours (min.)
 - j. energy density, 100 watt hours/pound
- 4) Mechanical Environmental Capability (design objectives):
 - a. Transportation Conditions. To be subjected to vibration and shock as per MIL-B-1 B Test 1 and perform as specified in electrical requirements.
 - b. Operation Environmental Conditions
 - 1) Mechanical Shock Test. The floor or barrier receiving the impact shall be of 2 inch fir backed by concrete or a rigid steel frame. The cell shall be dropped from a height of 48 inches. It shall be dropped on each face and, at 60° intervals around the circumference, on each corner and edge, a total of 20 drops. The drops shall be made from

a quick release hook or pendulum tester as made by the L. A. B. Corp. Skaneteles, N. Y., or equal.

- 2) Vibration Test. The cell shall be rigidly clamped to the platform of a vibration machine. A simple harmonic motion having an amplitude of 0.03 inch (0.06 inch total max. excursion) shall be applied. The frequency shall be varied either linearly at the rate of 1 cycle per second per minute or logarithmically between the limits of 10 and 55 cycles per second. The entire range of frequencies and return shall be traversed in not less than 90 minutes nor more than 100 minutes. Each cell shall be vibrated through the above mentioned frequency range in each of three mutually perpendicular directions.
- 3) Bounce Test. The cell shall be tested on the tester, type 1000-8C as made by the L. A. B. Corp., Skaneteles, N. Y., or equal.
 - a) Cover the tester bed with a panel of 1/2 inch plywood, with the grain parallel to the drive chain. Space six penny nails, with the heads below the surface, at 6 inch intervals around all four edges and at 3 inch intervals in a 6 inch square in the center.
 - b) Place the cell on the bed of the package tester. Limit the lateral motion of wooden fences, to not more than 3 inches and not less than 1 inch. Additional barriers may be used to prevent tumbling, provided that the fore-and-aft motion of the equipment against the back-stop is not restrained.
 - c) Operate the package tester, shafts in phase, for a total of 3 hours at 284 ± 2 rpm.

APPENDIX II

EQUIPMENT LIST

The following items of equipment were purchased under this contract.

1. Cathode molding dies (see Figure 5) 2.4" dia. cavity 1" deep
Supplier: Fen-Bar Precision Machinists, 9 Legion Drive,
Valhalla, New York.
2. Steel Rule Die - 2-1/4" dia. for current collectors
Supplier: Progressive Steel Rule Die, 10 Alberton St., Lynn, Mass.
3. Cold Weld Press Frame (see Figure 6) 12 ton capacity
Supplier: Mill Lane Engineering, 12 Garfield Circle, Burlington, Mass.
4. Cold Welding Dies (see Figure 6) for closing 2.55 dia. can
Supplier: Scully Jones Div. Bendix Corp., 1901 S. Rockwell St.,
Chicago, Illinois
5. Hydraulic Cylinder, RD-96 Pump and P84 Accessories, 12 ton and
6" stroke for cold weld press
Supplier: R. H. Scales, 121 Brookline Ave., Boston, Mass.
6. Steel rule die 2-27/64" dia. for cell separators
Supplier: Progressive Steel Rule Die Co., 10 Alberton St., Lynn, Mass.

MATERIAL LIST

- | | | |
|------------------------|--------------------|---------------------------------------|
| 1. Can | - LPS-35-C-1-1169 | Roland-Tiener Co.
Everett, Mass. |
| 2. Cover | - LPS-35-C-2-1169 | Roland-Tiener Co.
Everett, Mass. |
| 3. Cathode collector | - LPS-35-CC-10-170 | Exmet Corp.,
Bridgeport, Conn. |
| 4. Anode collector | - LPS-35-AC-11-270 | Exmet Corp.,
Bridgeport, Conn. |
| 5. Glass to metal seal | - LPS-35-GM-1-1269 | Glasseal Corp.,
Linden, N. J. |
| 6. Separator | - LPS-35-S-1-270 | Scientific Products
Waltham, Mass. |

APPENDIX III

TABLE 1: Constant Power Discharge (Pulse) Currents for 35AH Cell

Power(watts)	Voltage (volts)	Current (amps)
5.00	1.50	3.33
5.00	1.60	3.12
5.00	1.70	2.94
5.00	1.80	2.77
5.00	1.90	2.63
5.00	2.00	2.50
5.00	2.10	2.38
5.00	2.20	2.27
5.00	2.30	2.17
5.00	2.40	2.08
5.00	2.50	2.00
5.00	2.60	1.92
5.00	2.70	1.85
5.00	2.80	1.78
5.00	2.90	1.72
5.00	3.00	1.66

TABLE 2: Constant Power Discharge (Continuous) Currents for 35AH Cells

Power (watts)	Voltage (volts)	Current (amps)
0.0150	1.5000	0.0100
0.0150	1.6000	0.0094
0.0150	1.7000	0.0088
0.0150	1.8000	0.0083
0.0150	1.9000	0.0079
0.0150	2.0000	0.0075
0.0150	2.1000	0.0072
0.0150	2.2000	0.0068
0.0150	2.3000	0.0065
0.0150	2.4000	0.0063
0.0150	2.5000	0.0060
0.0150	2.6000	0.0058
0.0150	2.7000	0.0056
0.0150	2.8000	0.0054
0.0150	2.9000	0.0052
0.0150	3.0000	0.0050

TABLE 3: Constant Power Discharge Pulse Currents Single Cathode 2.4" Diameter Cells. (CD equivalent to CD for 5 watts on 13 cathode 35AH cell).

Volts	Amps
1.50	0.256
1.60	0.240
1.70	0.226
1.80	0.213
1.90	0.202
2.00	0.192
2.10	0.183
2.20	0.175
2.30	0.167
2.40	0.160
2.50	0.154
2.60	0.147
2.70	0.142
2.80	0.136
2.90	0.132
3.00	0.128

TABLE 4: Constant Power Continuous Discharge Currents Single Cathode 2.4" Diameter Cells: (CD equivalent to CD for 15 milliwatts on 13 cathode 35AH cells).

Volts	Milliamperes
1.50	0.77
1.60	0.72
1.70	0.67
1.80	0.63
1.90	0.61
2.00	0.58
2.10	0.55
2.20	0.52
2.30	0.50
2.40	0.48
2.50	0.46
2.60	0.45
2.70	0.43
2.80	0.42
2.90	0.40
3.00	0.38

TABLE 5: Constant Power Discharge Pulse Currents Single Cathode
2.4" Diameter Cells (CD equivalent to CD for 5 watts on
20 cathode cell).

Volts	Pulse Amp
1.50	0.167
1.60	0.157
1.70	0.147
1.80	0.138
1.90	0.131
2.00	0.125
2.10	0.119
2.20	0.113
2.30	0.109
2.40	0.104
2.50	0.100
2.60	0.096
2.70	0.093
2.80	0.089
2.90	0.086
3.00	0.083

TABLE 6: Constant Power Continuous Discharge Currents Single Cathode
2.4" Diameter Cells (CD equivalent to CD for 15 milliwatts on
20 cathode cell).

Volts	Milliamperes
1.50	0.500
1.60	0.470
1.70	0.440
1.80	0.415
1.90	0.395
2.00	0.375
2.10	0.360
2.20	0.340
2.30	0.325
2.40	0.315
2.50	0.300
2.60	0.290
2.70	0.280
2.80	0.270
2.90	0.260
3.00	0.250

TABLE 7: Circular Cell Pulse Test Summary

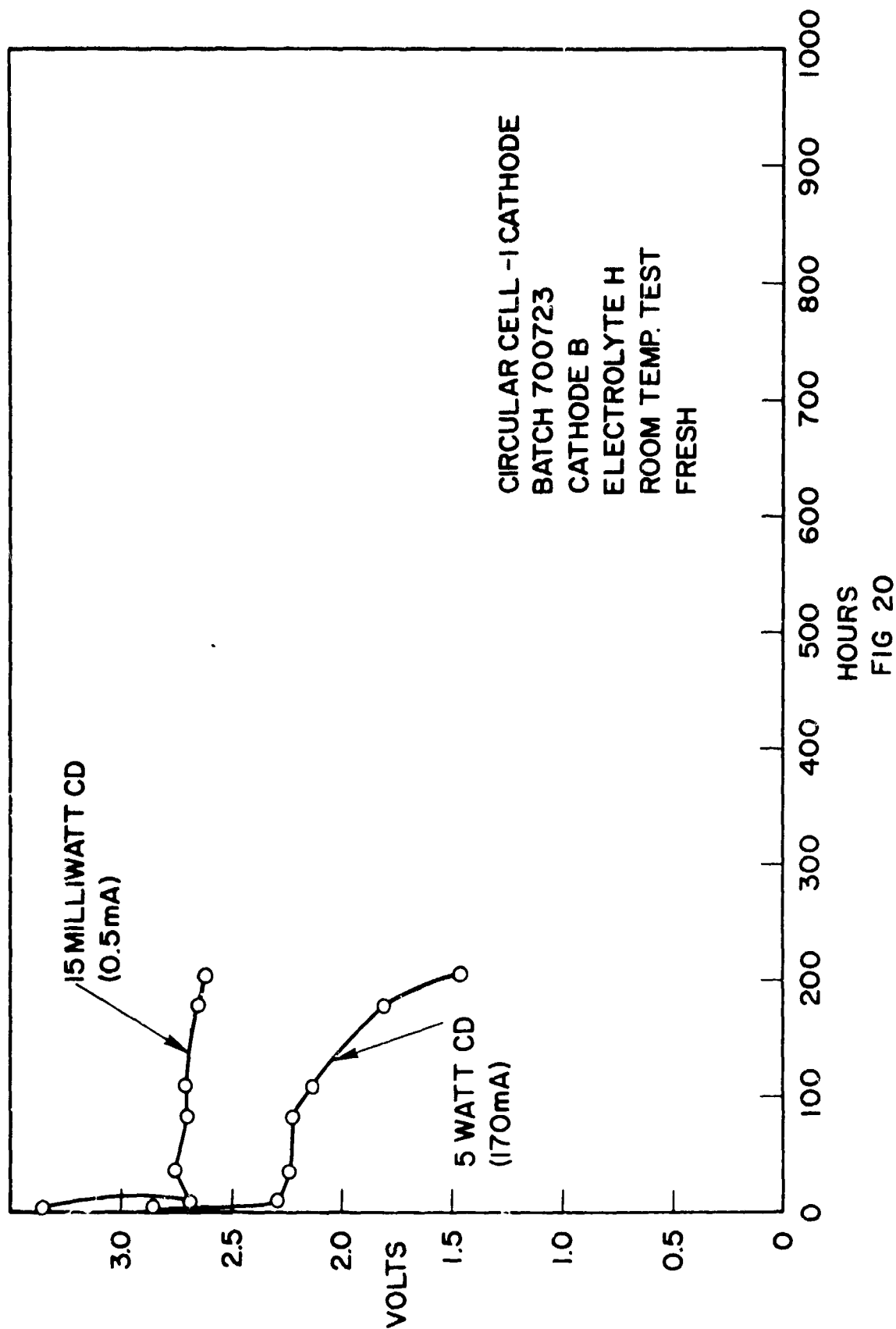
Batch No.	Cathode Type	Electrolyte Type	Storage Time & Condition	Test Temperature °C	Extrapolated Energy Density whr/lb to 1.5 volt	Discharge Fig. No.	Notes
700723	B	H	Fresh	Room	19.6		Constant Current
700723	B	H	Fresh	-30	43.5		Constant Current
700723	B	H	55°C	---	---	---	Destroyed
700807	B	J	Fresh	Room	97.4		Constant Power
700807	B	J	Fresh	-30	40.8		Constant Power
700807	B	J	55°C	---	---	---	Destroyed
700811	B	K	Fresh	-30	42.8		Constant Power
700811	B	K	3 mo. Room	-30	65		Constant Power
700811	B	K	3 mo. 45°C	-30	40		Constant Power
700811	B	K	3 mo. 55°C	-30	--		Discontinued
701010	B	R	Fresh	Room	96		Constant Power
701010	B	R	Fresh	-30	54		Constant Power

TABLE 8: 35AH Cell Pulse Test Summary

Batch No.	Cathode Type	Electrolyte Type	Storage Time & Condition	Test Temperature °C	Energy Density whr/lb	Discharge Fig. No.	Notes
700529	B	H	Fresh	Room	27		Constant Current
700601	B	H	Fresh	-30	Discontinued		Constant Current
700618	B	H	Fresh	Room	50		Constant Power
700618	B	H	Fresh	-30	40		Constant Power
700806	B	H	Fresh	Room	55		Constant Power
700806	B	H	Fresh	-30	27		Constant Power
700821	B	H	Fresh	Room	60		Constant Power
700821	B	H	Fresh	-30	33		Constant Power

TABLE 9: 35AH Cell Pulse Test Summary, 20 Cathode/Cell

Batch No.	Cathode Type	Electrolyte Type	Storage Time & Condition	Test Temperature °C	Energy Density whr/lb	Discharge Fig. No.	Notes
700923	B	H	Fresh	Room	89		10% Duty Cycle
700928	B	R	Fresh	Room	84.5		10% Duty Cycle
700928	B	R	Fresh	-30	22		10% Duty Cycle
700928	B	R	1 wk @ 55°C	Room	57		10% Duty Cycle
700928	B	R	1 wk @ 55°C	-30	13		10% Duty Cycle
701008	B	R	Fresh	Room	72.5		10% Duty Cycle
701012	B	R	Fresh	Room	102		10% Duty Cycle
700928	B	R	After Vibration	Room	80		10% Duty Cycle



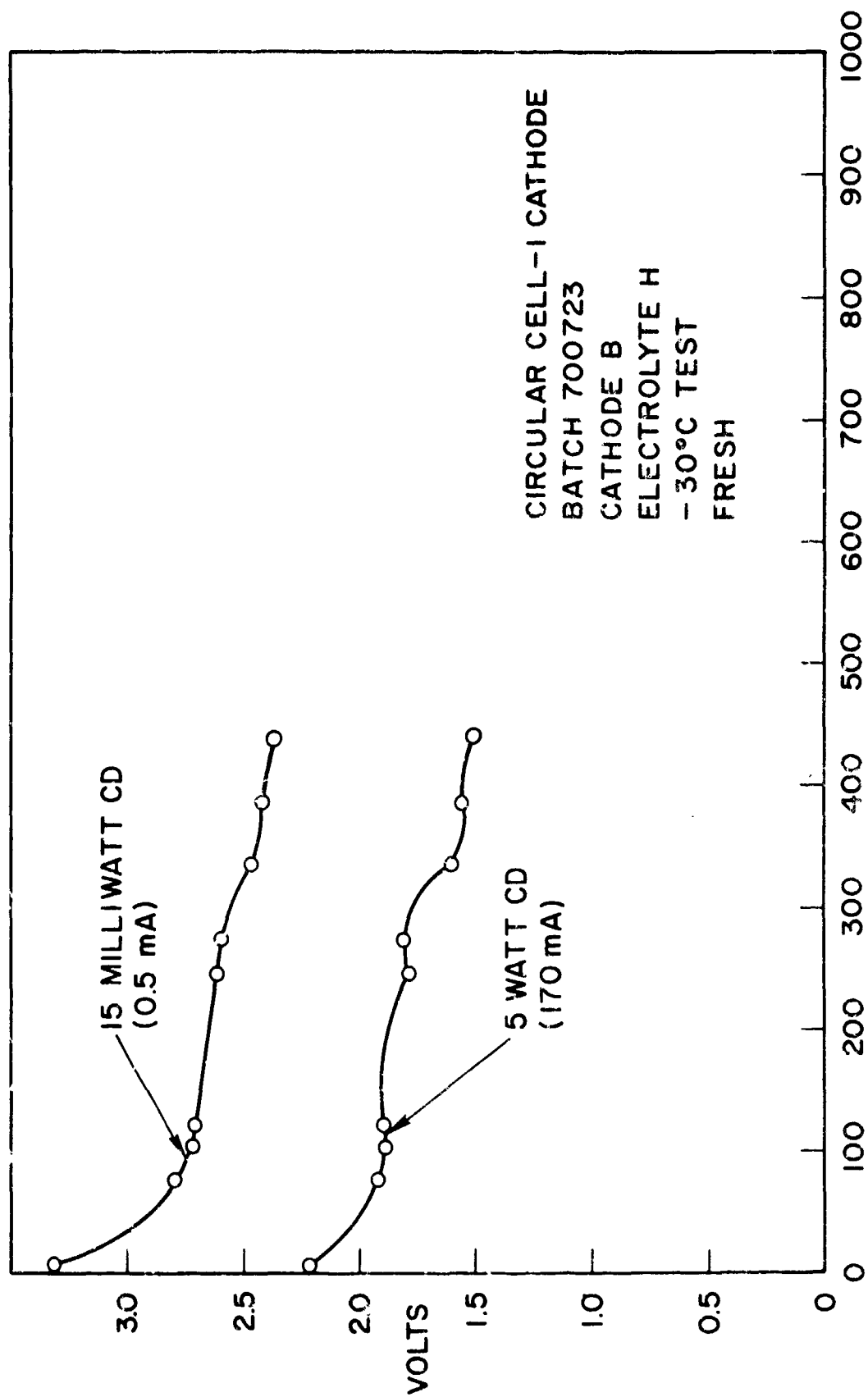
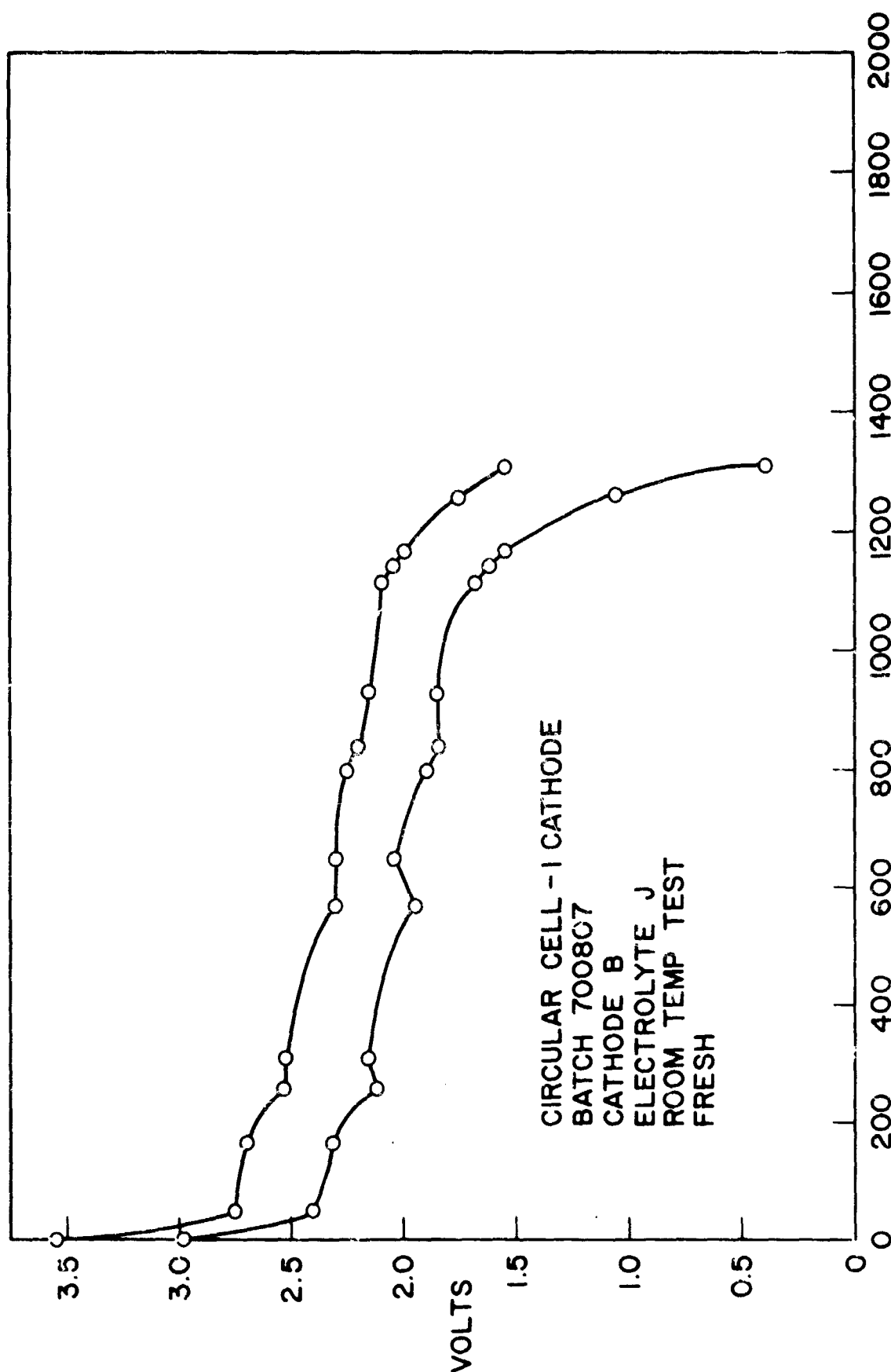


FIG. 21



CIRCULAR CELL - I CATHODE
BATCH 700807
CATHODE B
ELECTROLYTE J
ROOM TEMP TEST
FRESH

FIG. 22

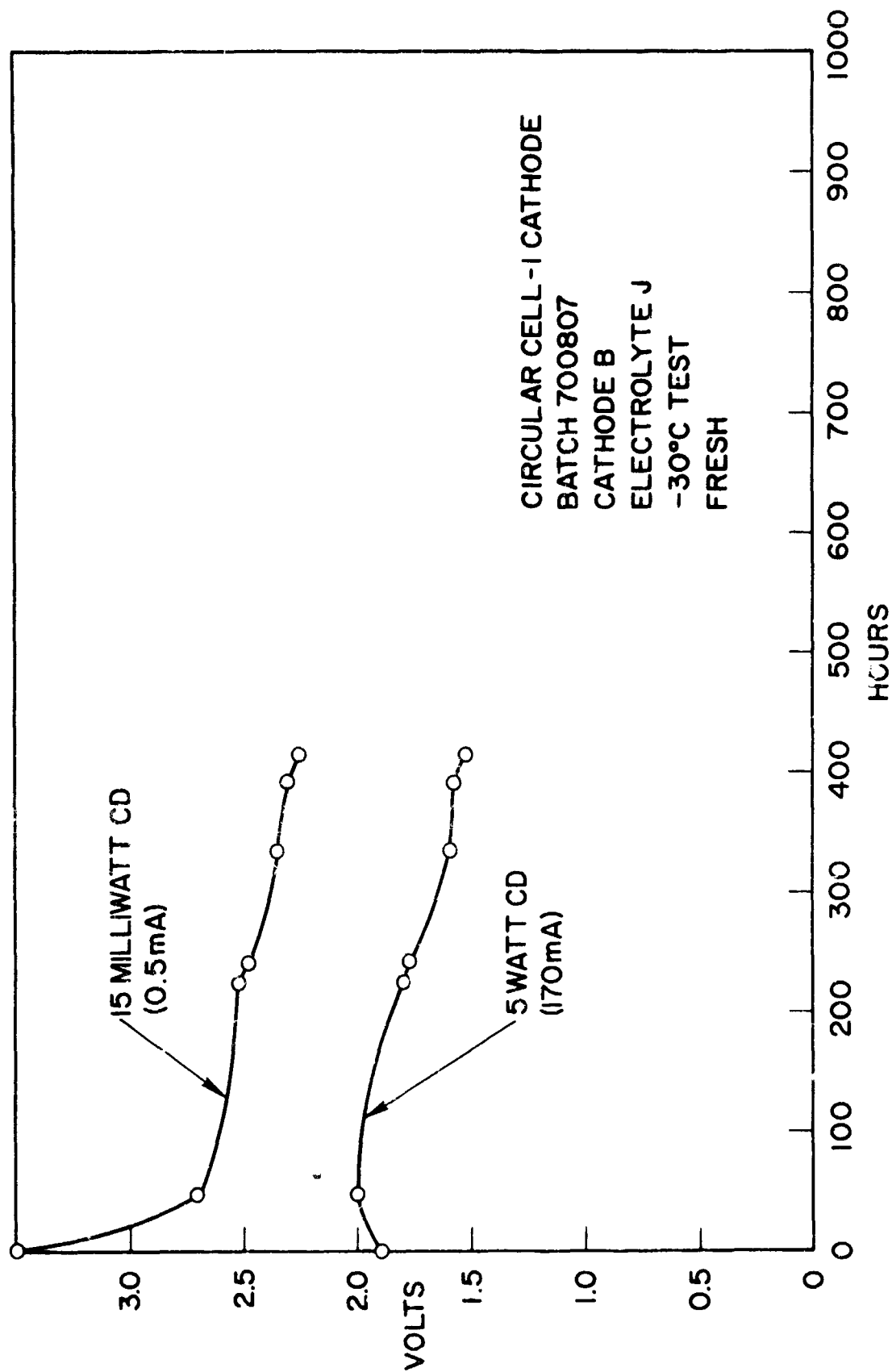


FIG 23

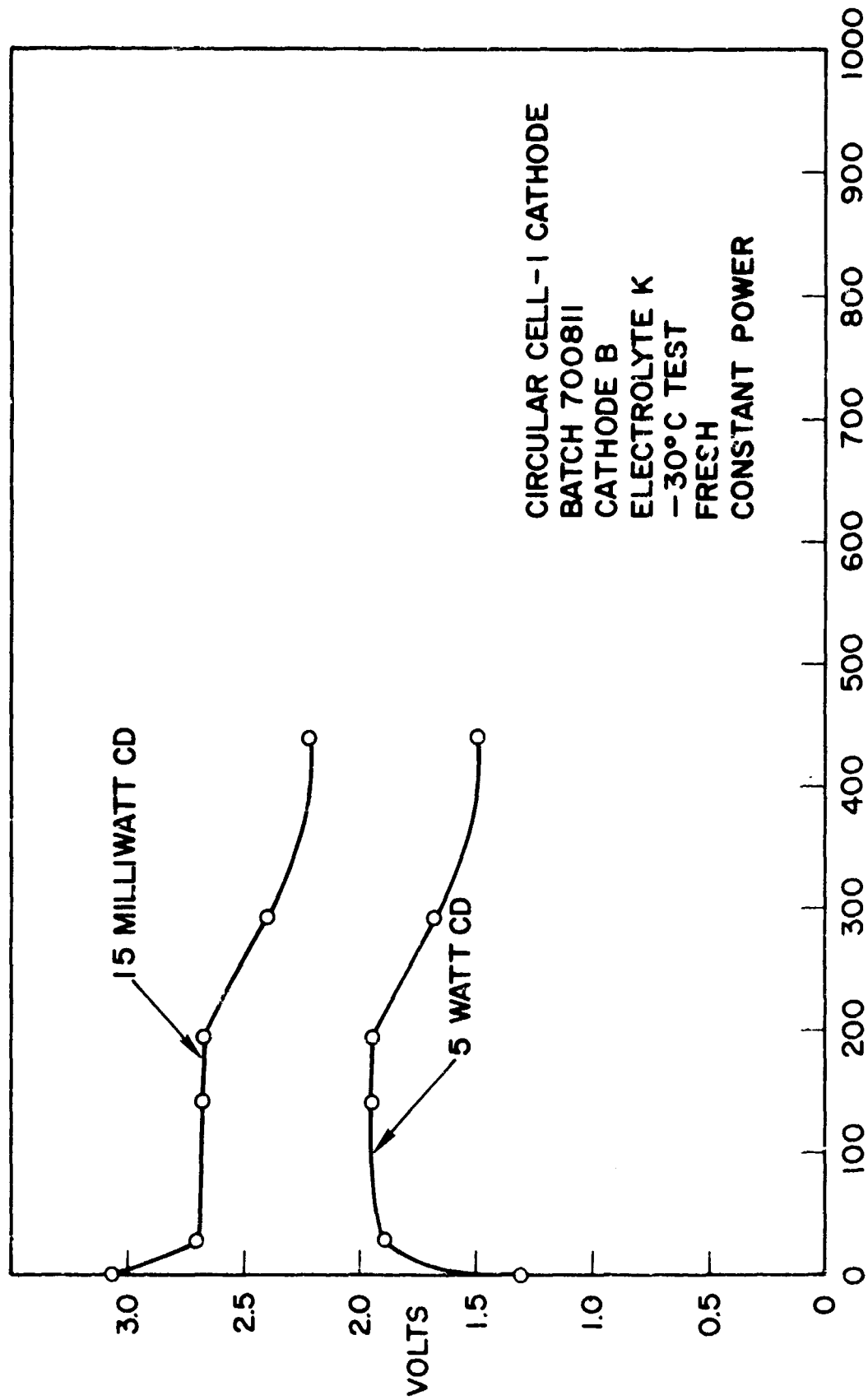


FIG. 24

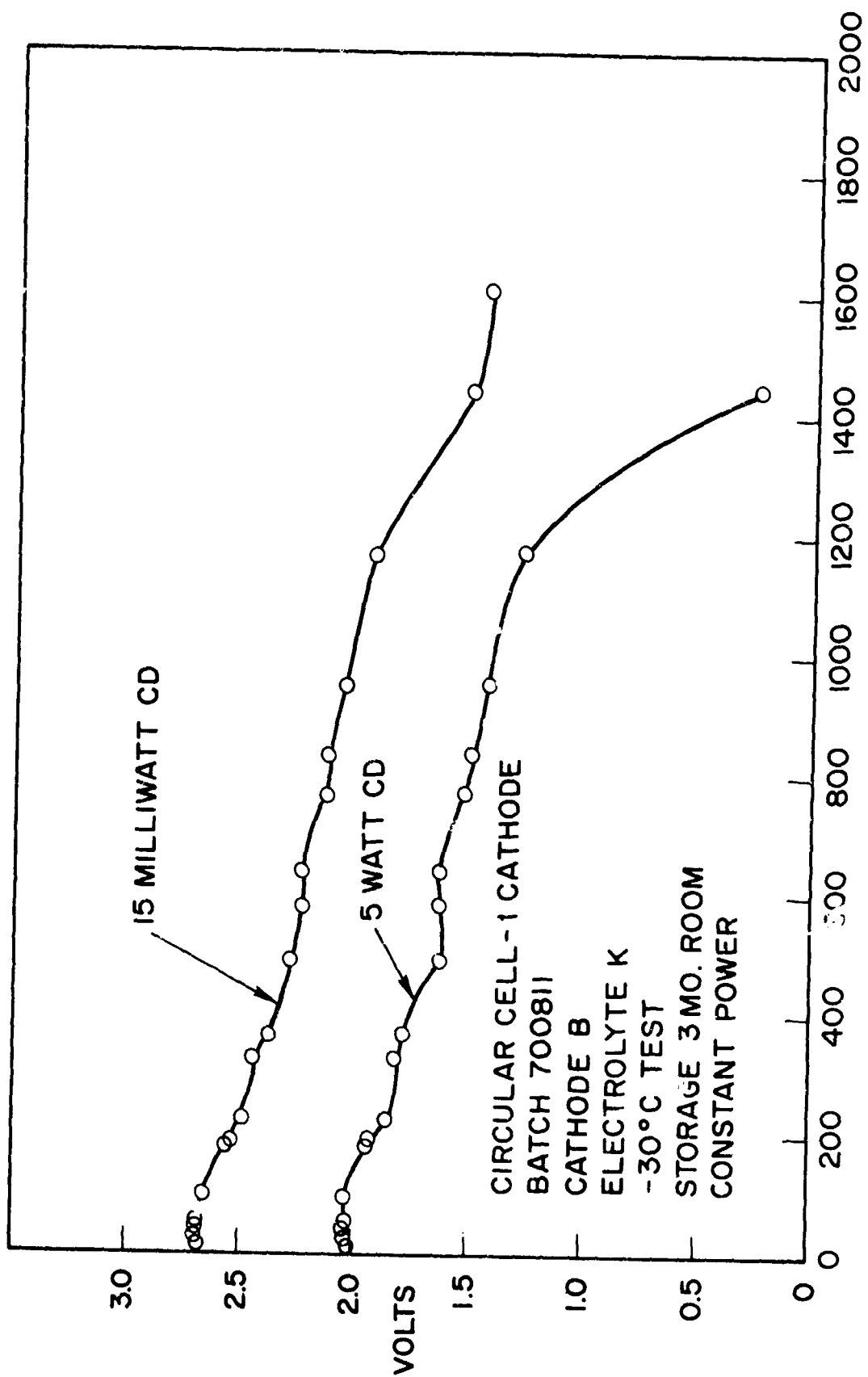


FIG. 25

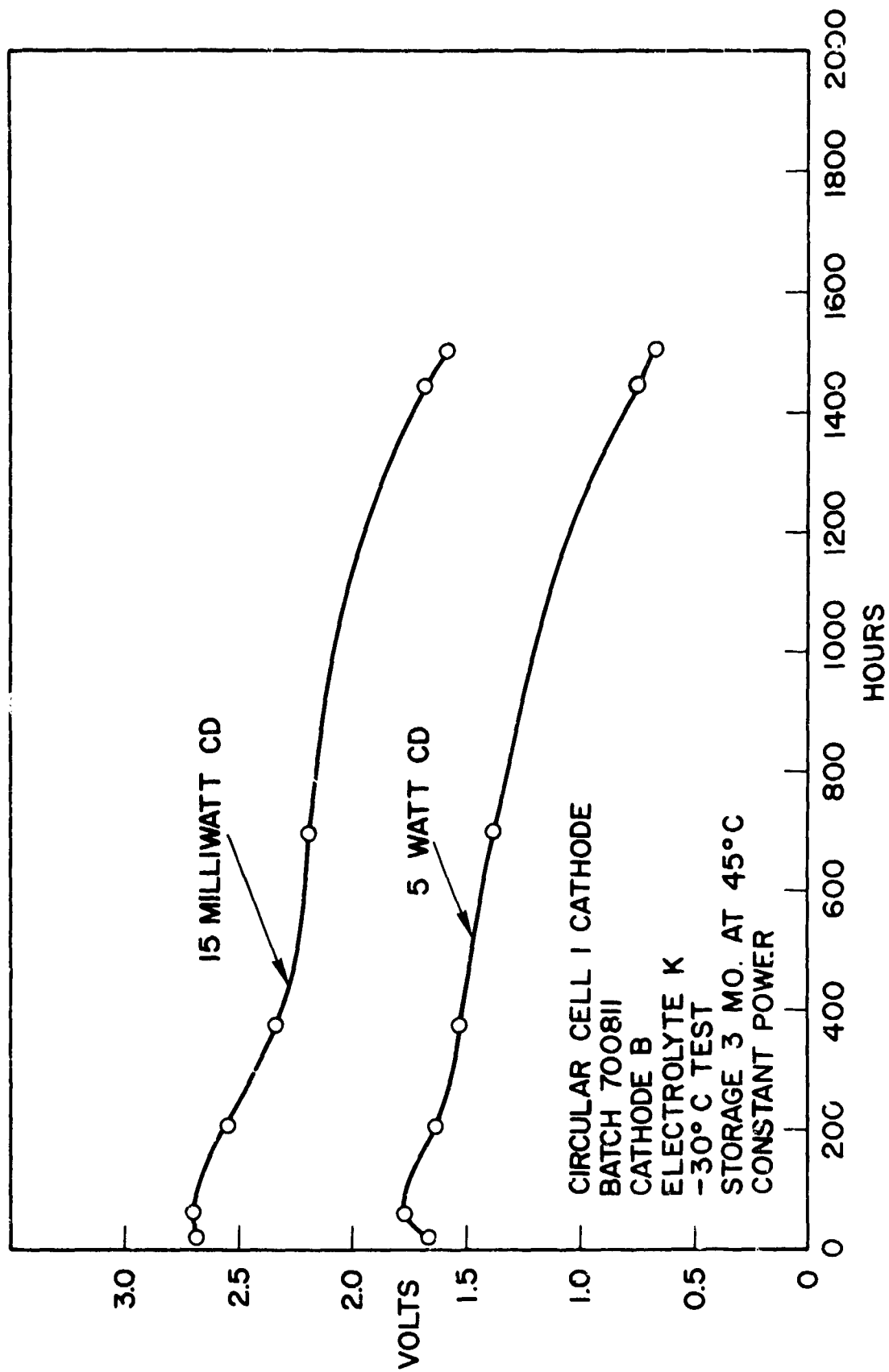
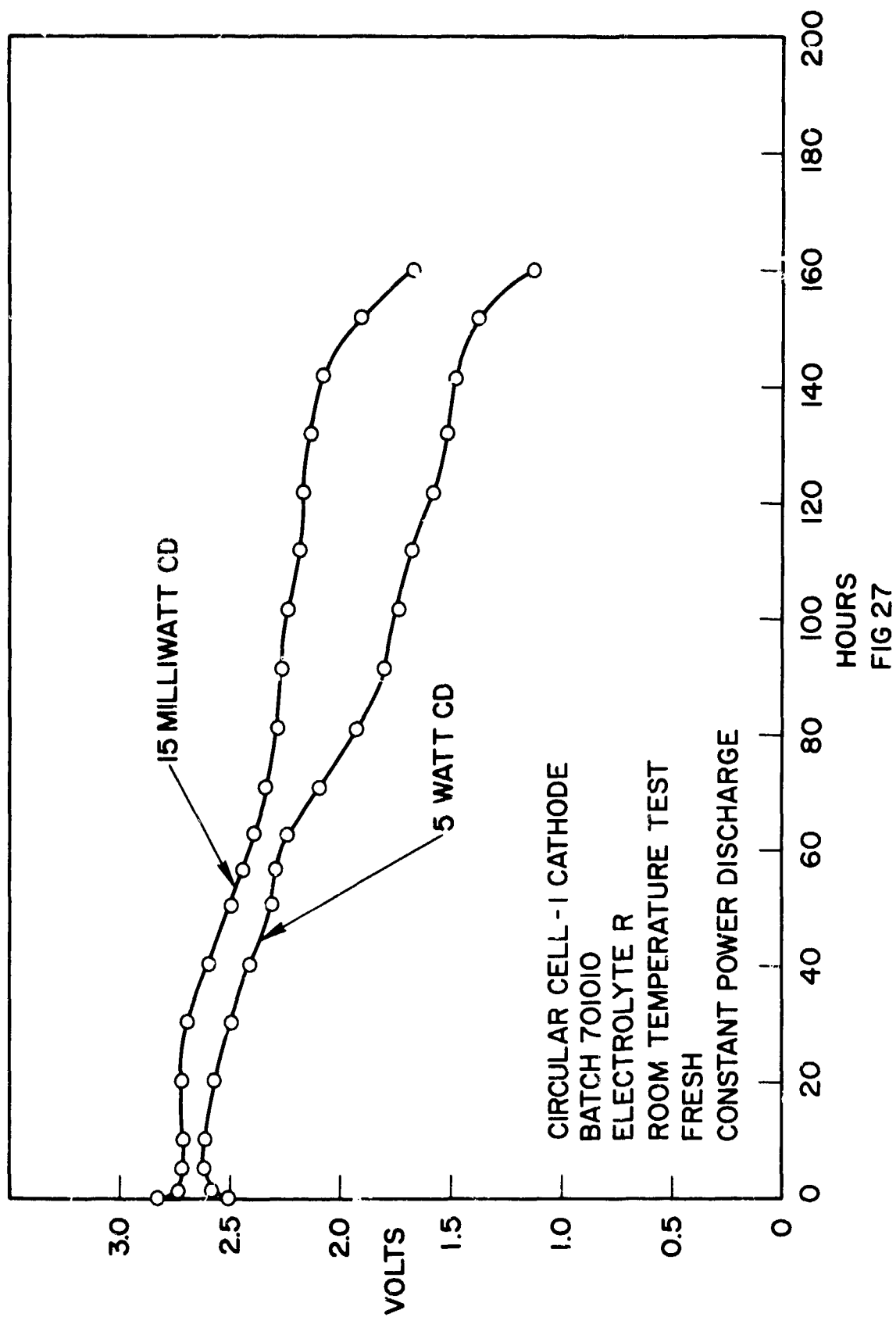
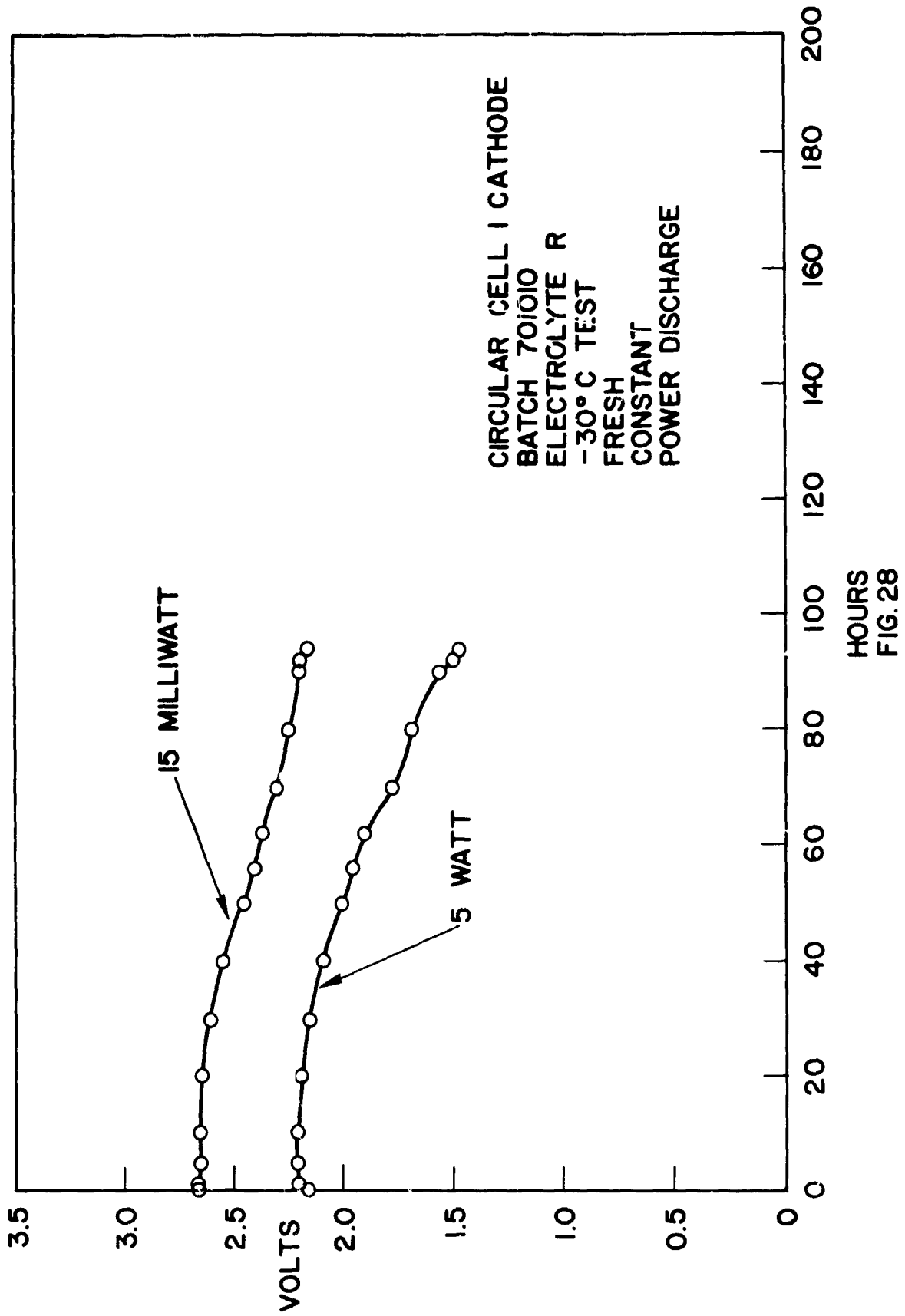


FIG. 26





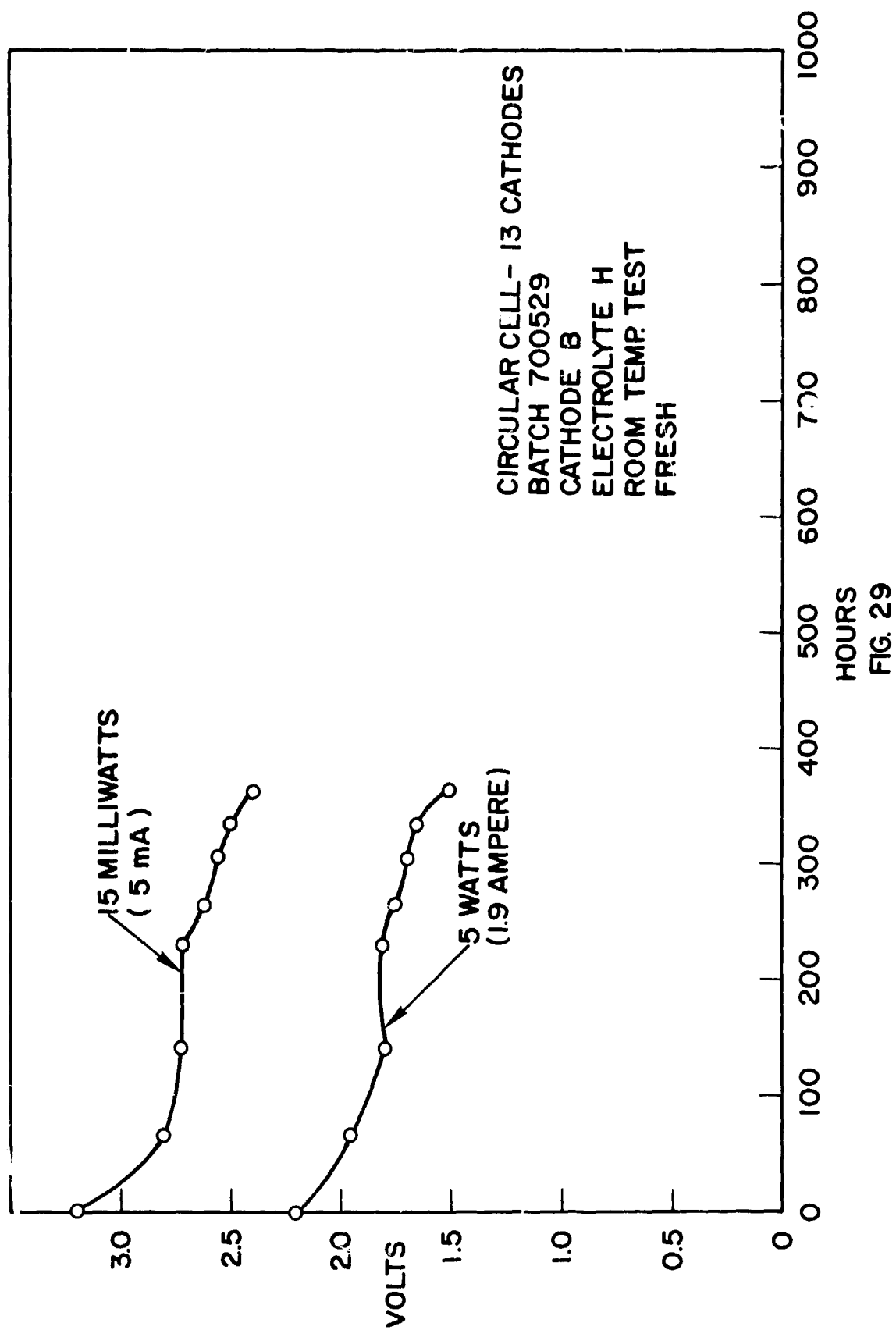
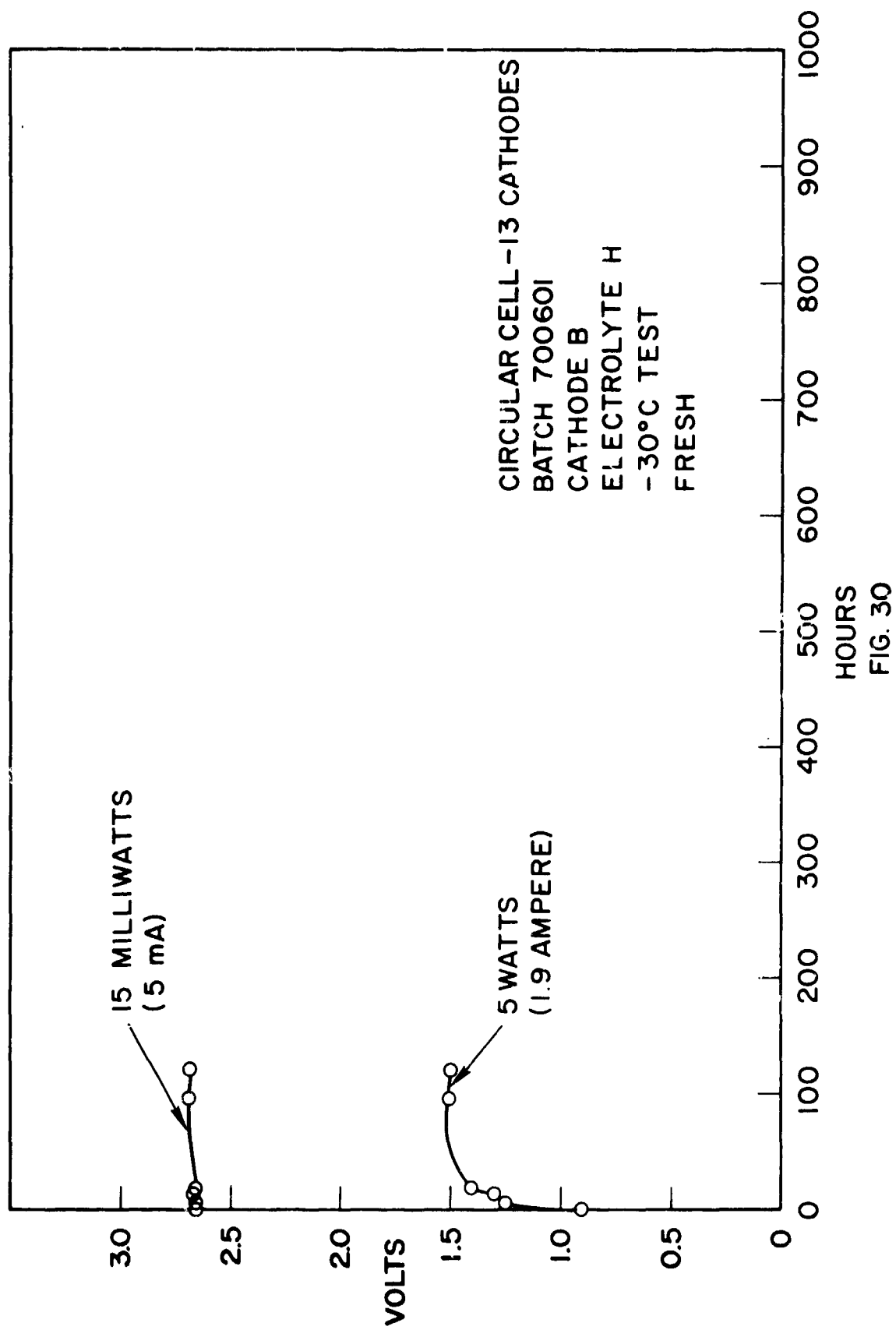


FIG. 29



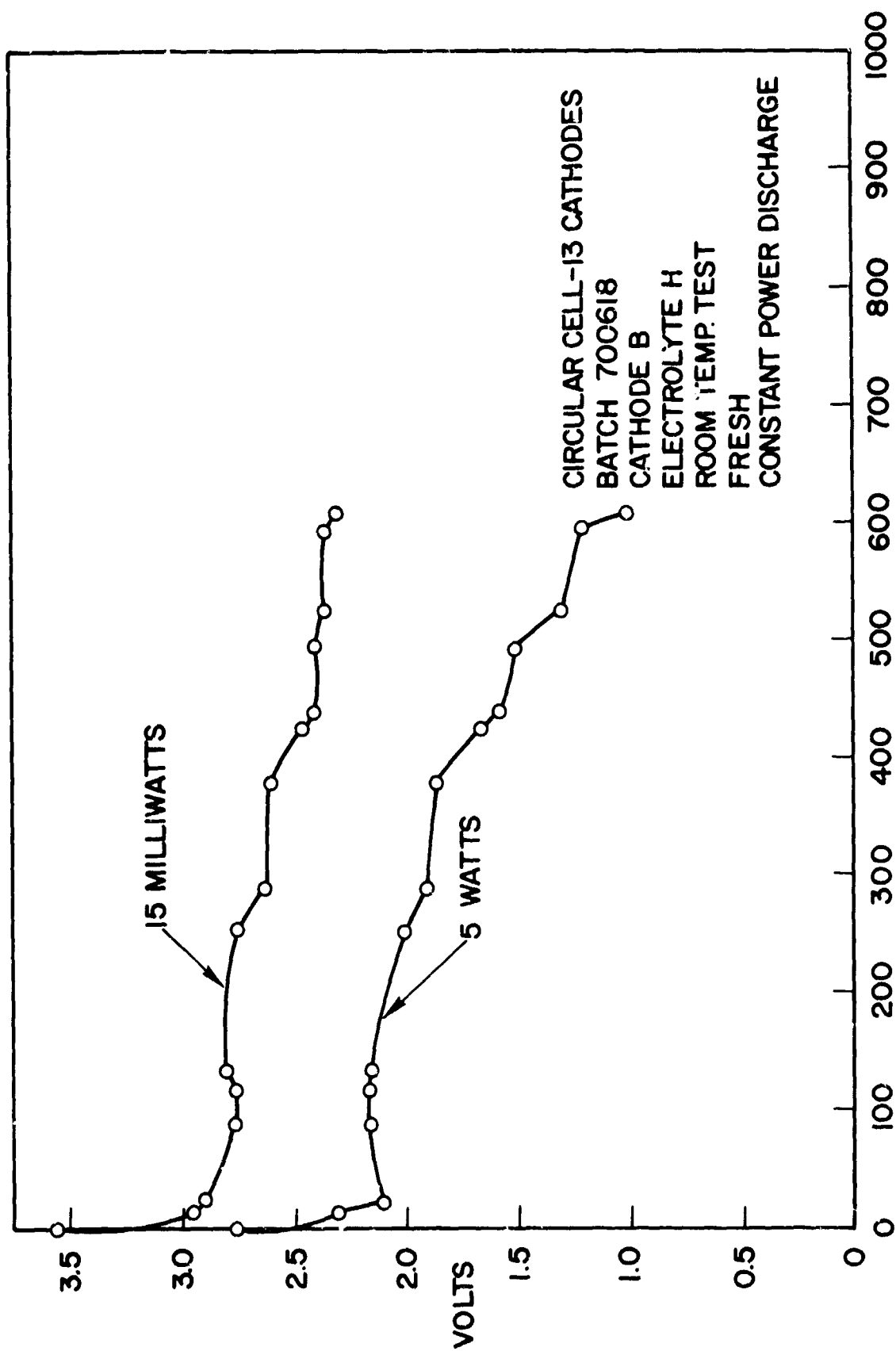


FIG. 31

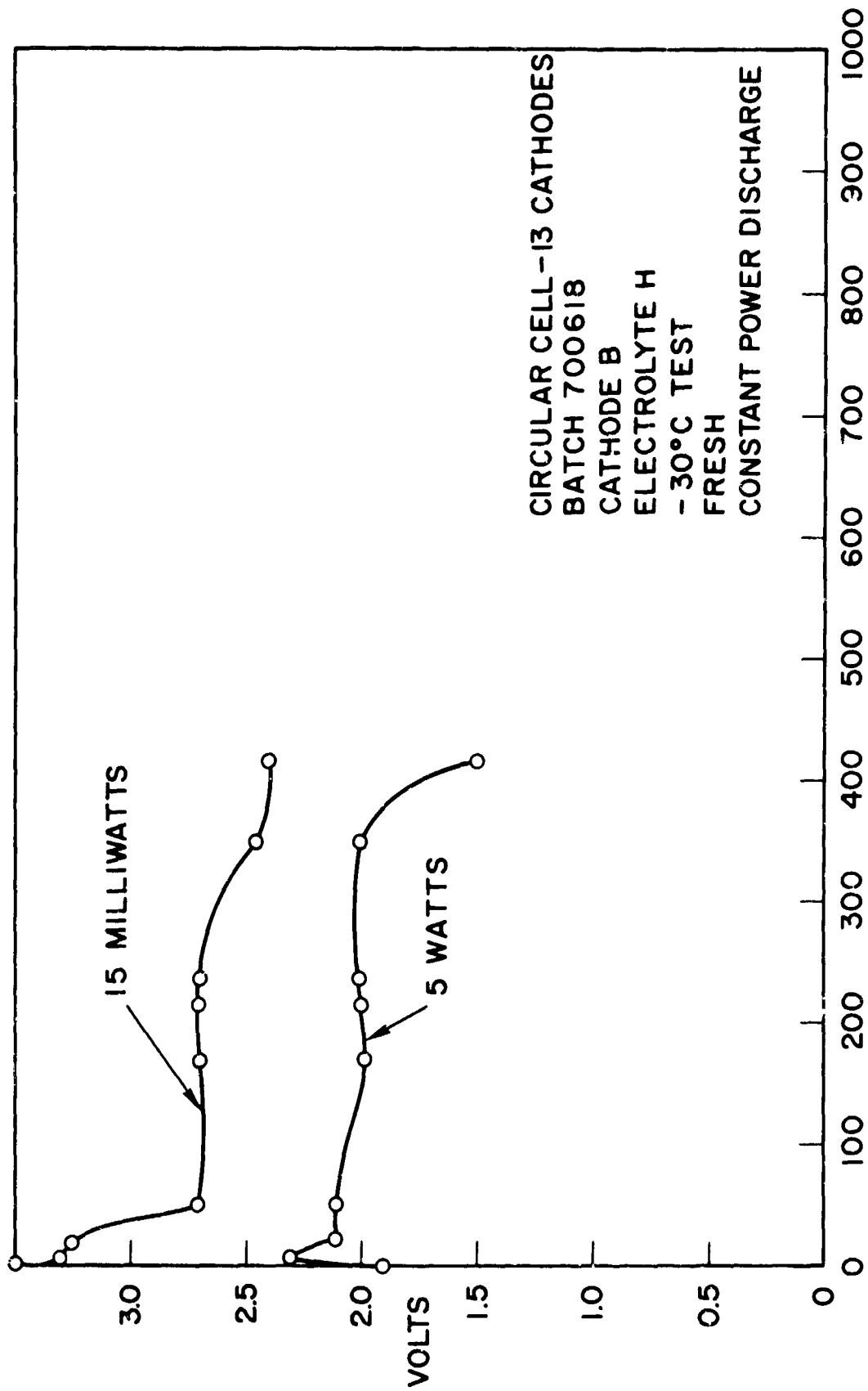


FIG. 32

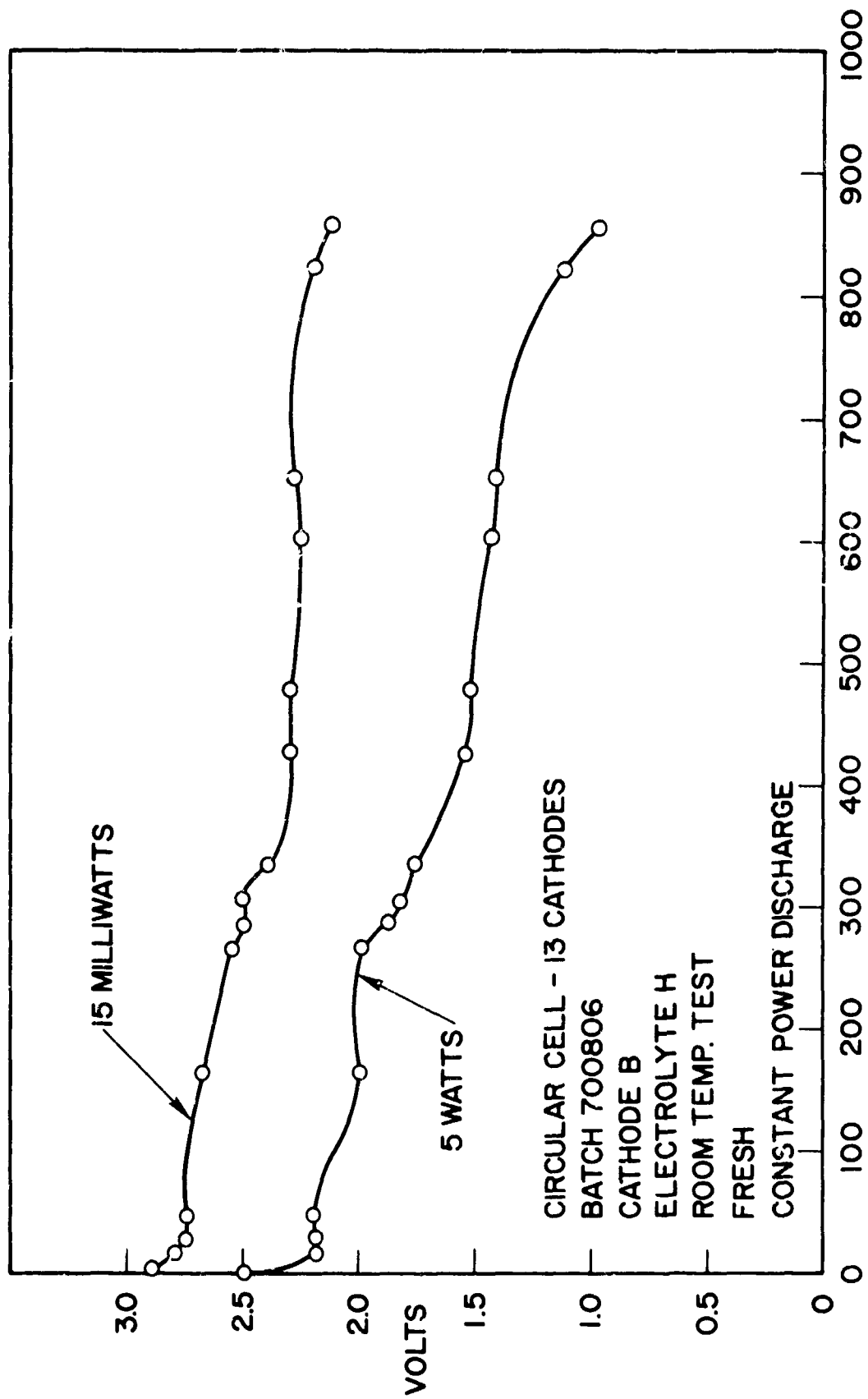


FIG. 33

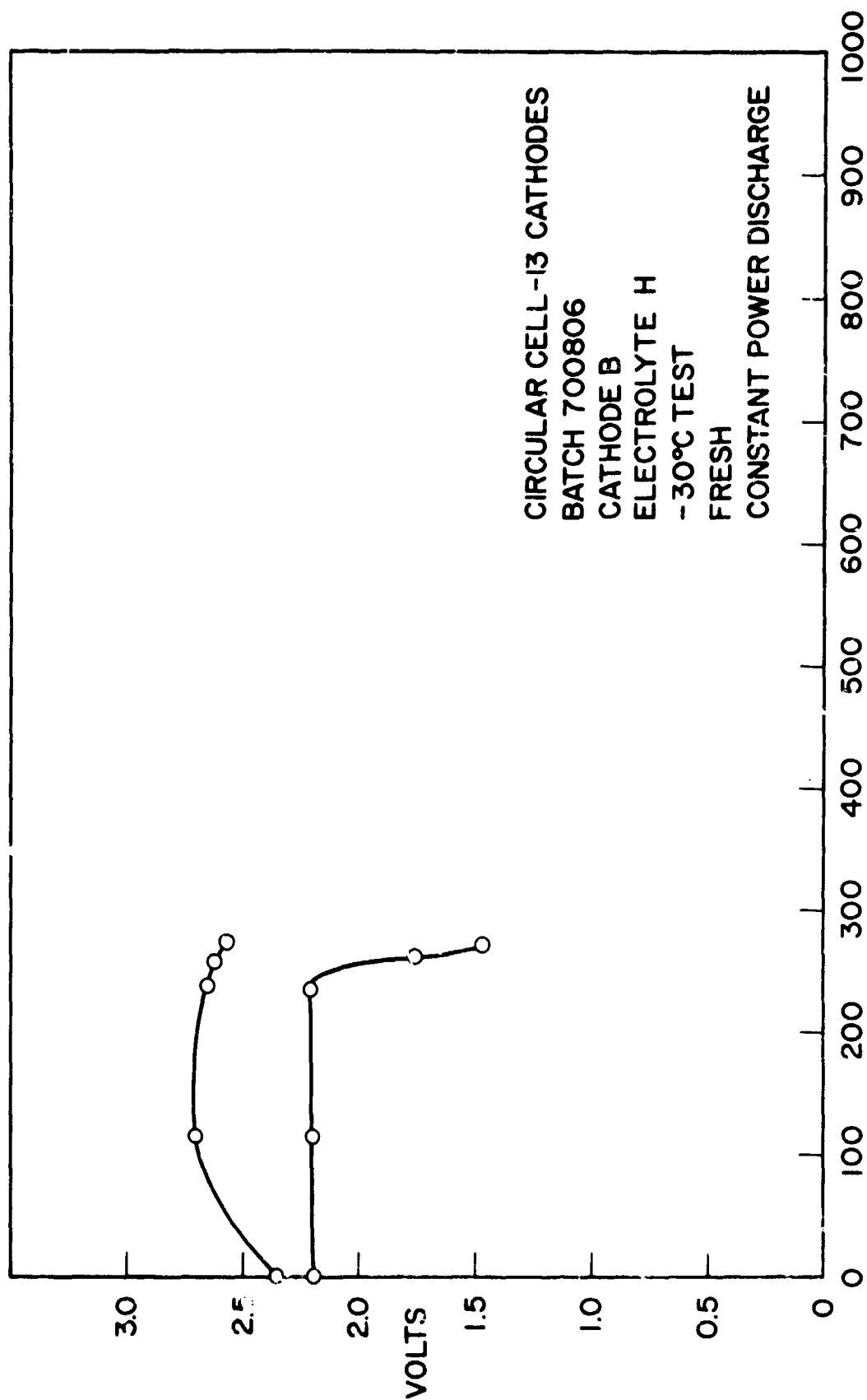


FIG 34

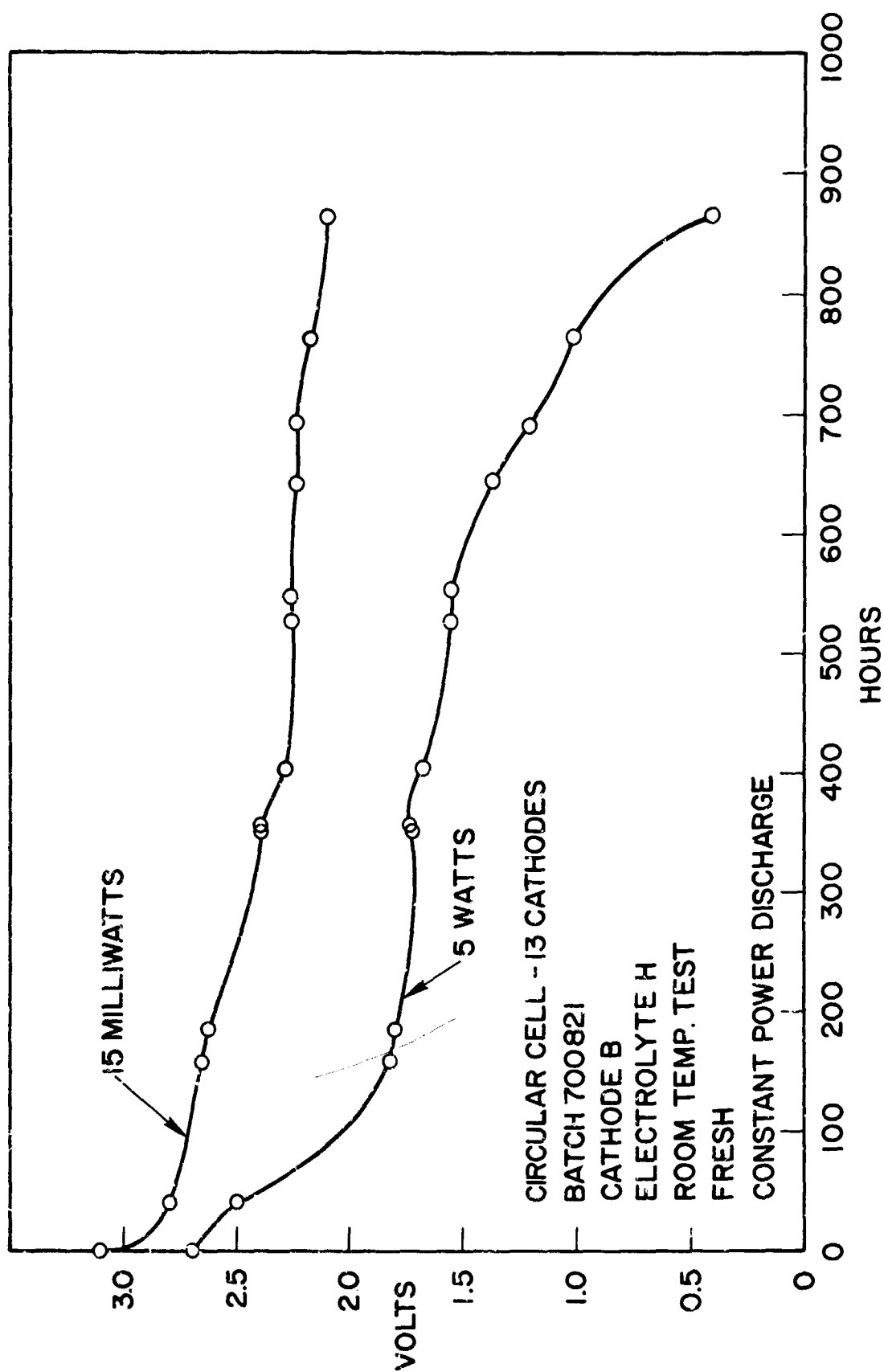


FIG. 35

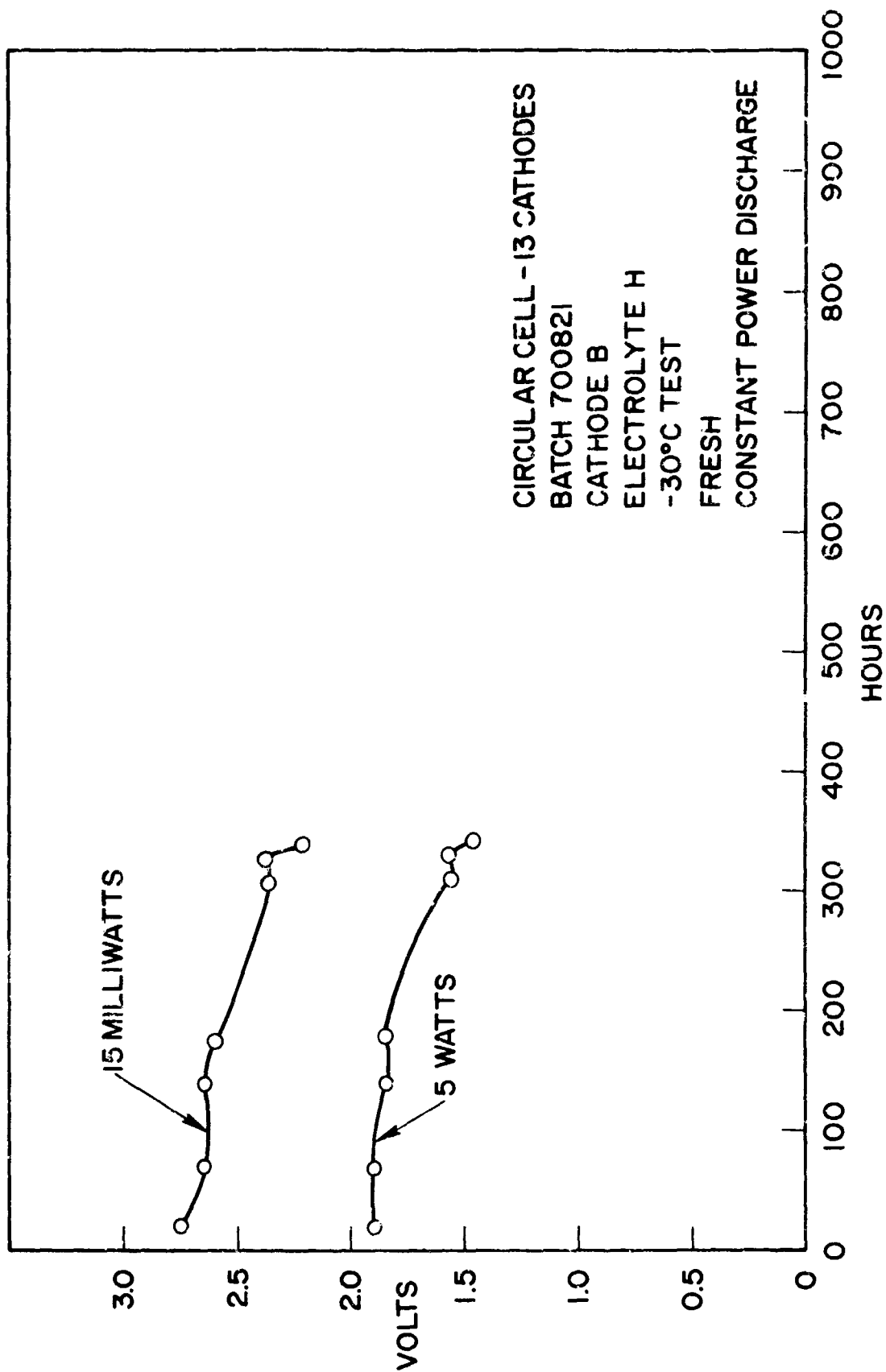


FIG. 36

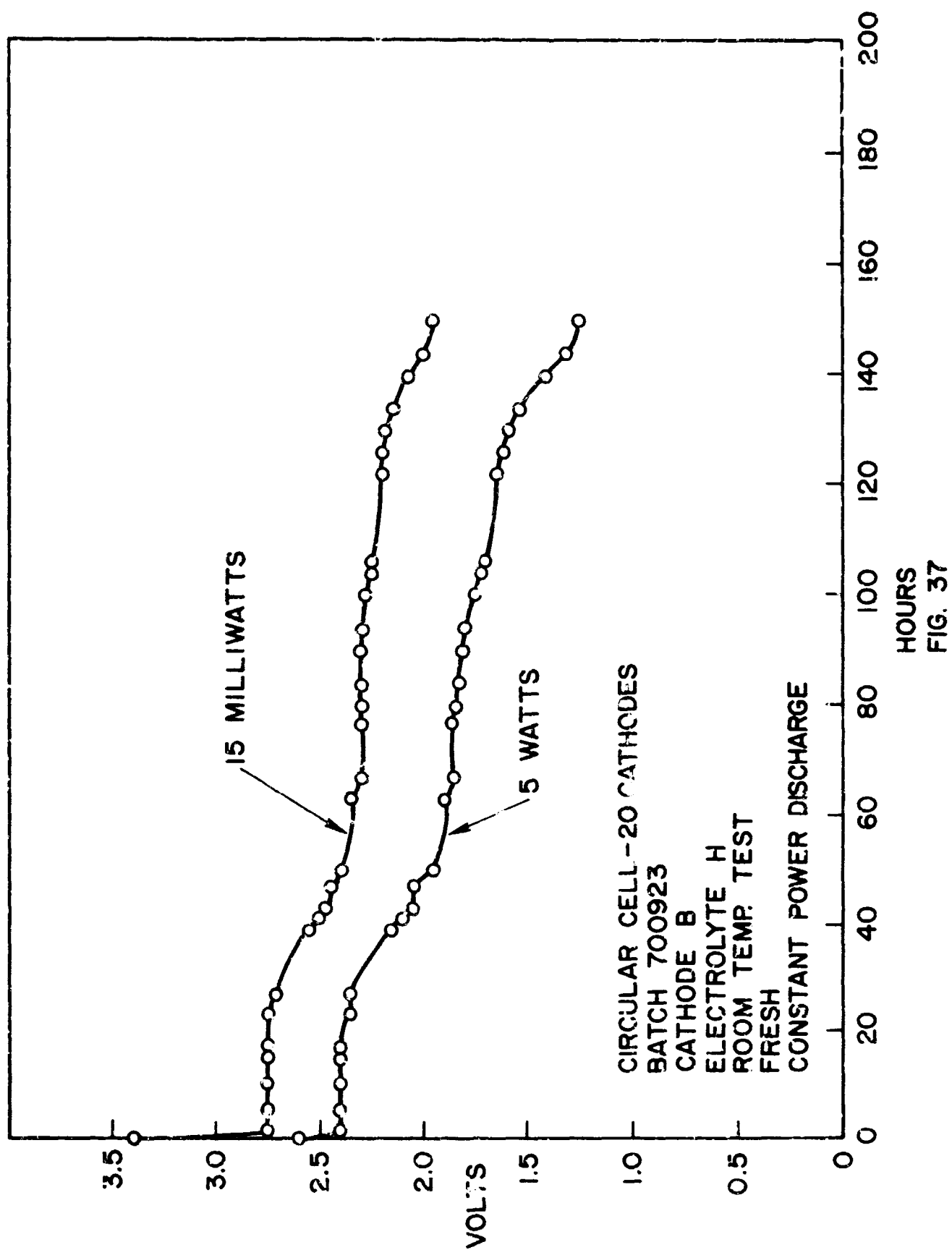
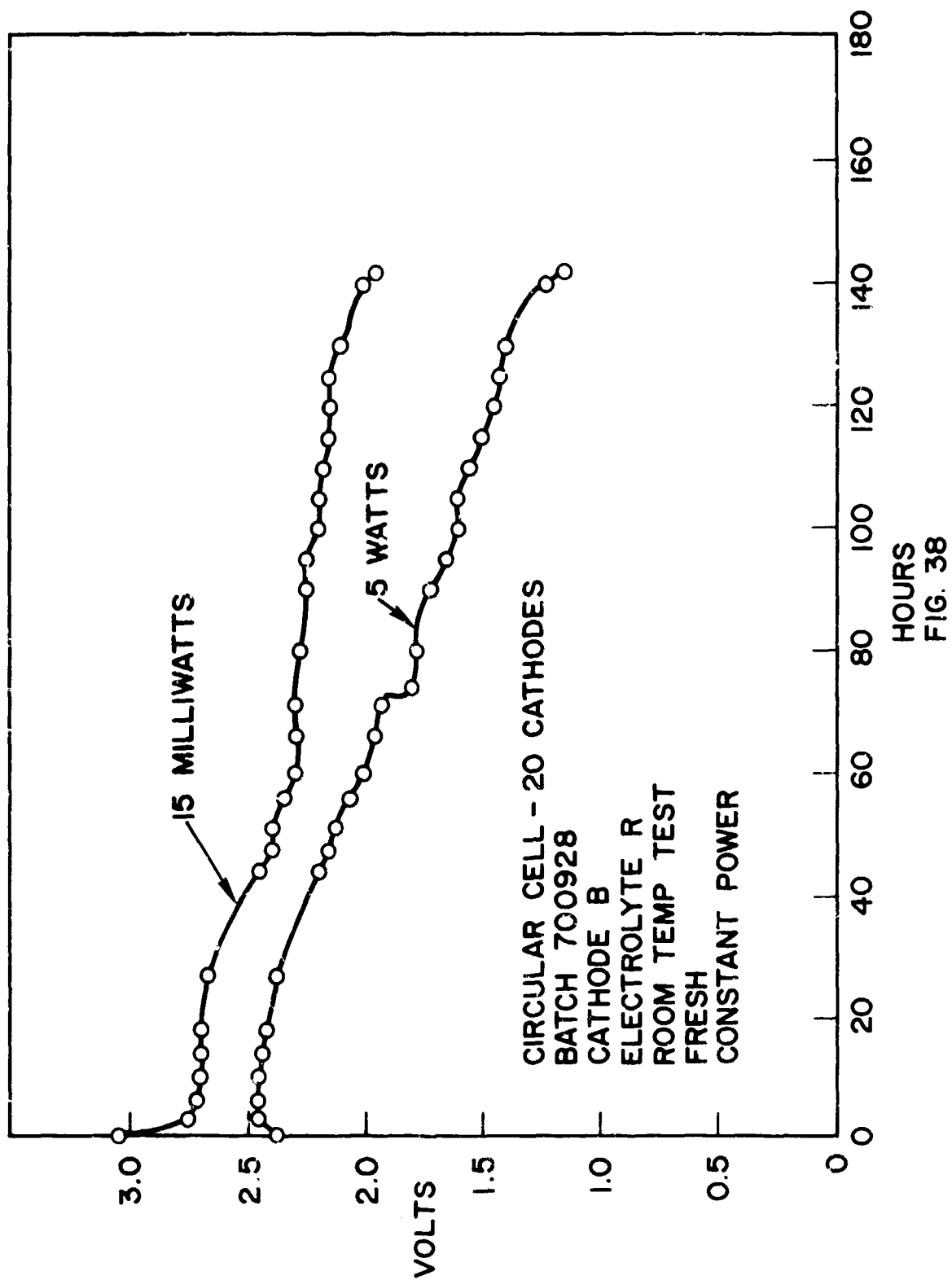
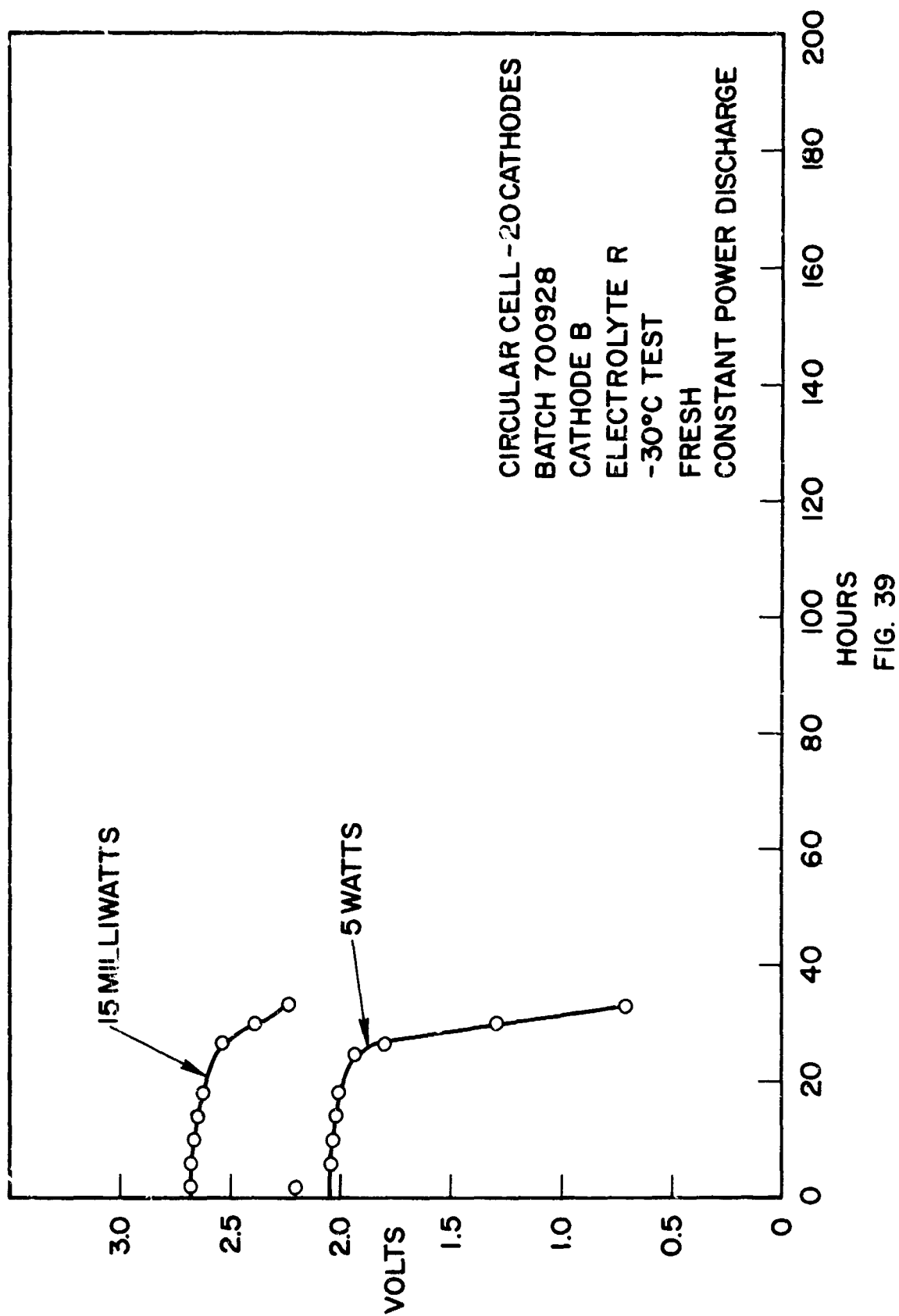


FIG. 37





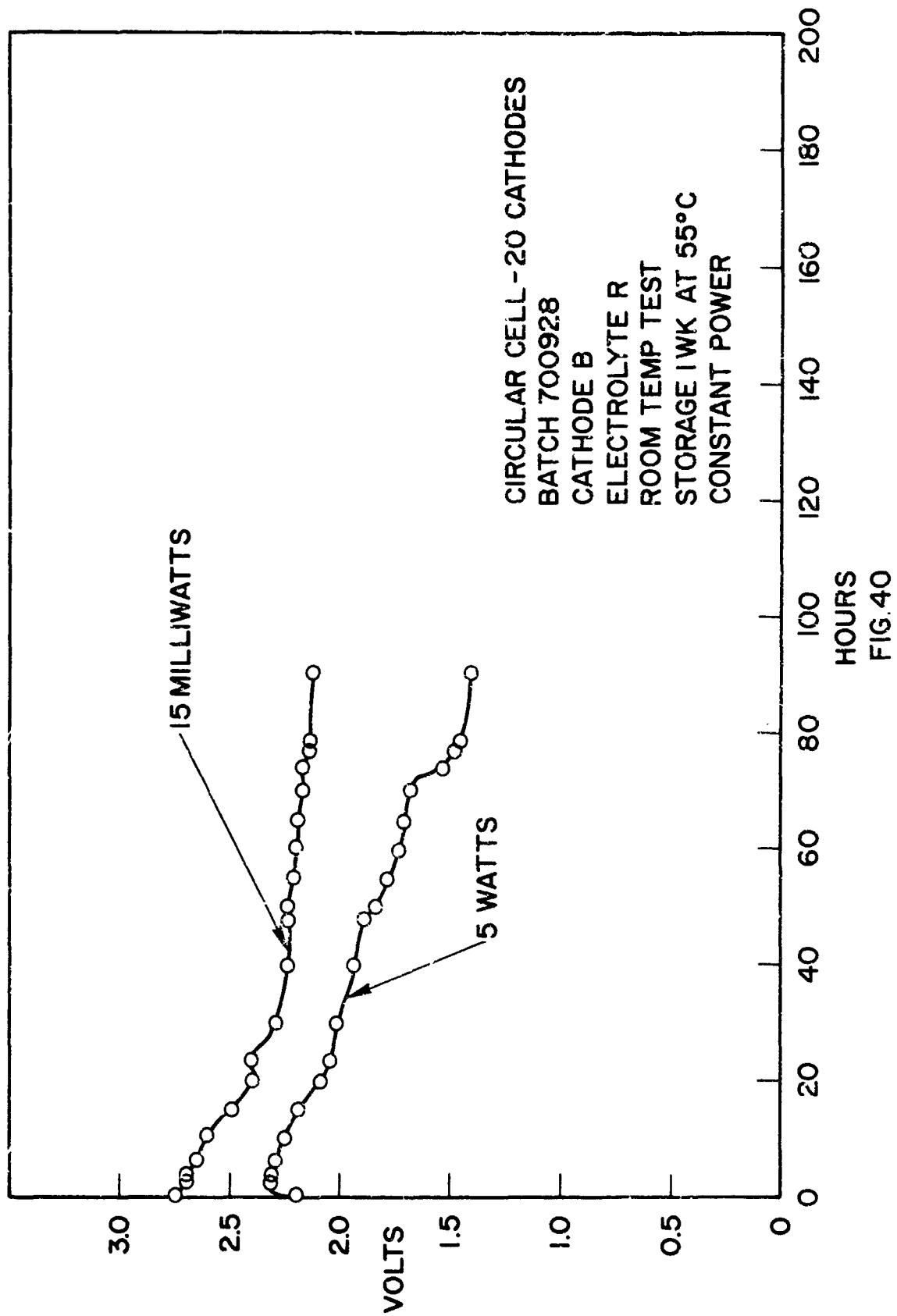
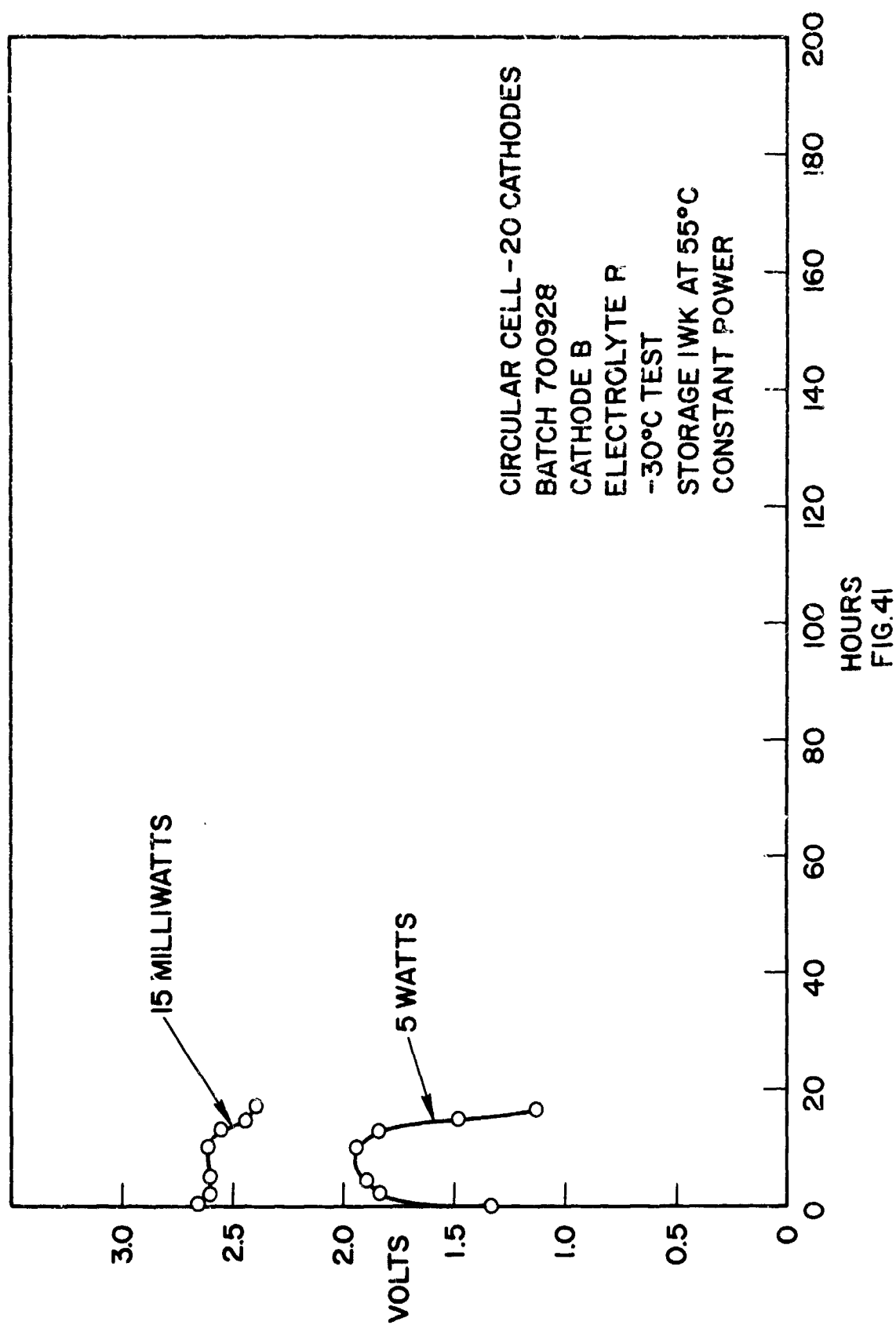
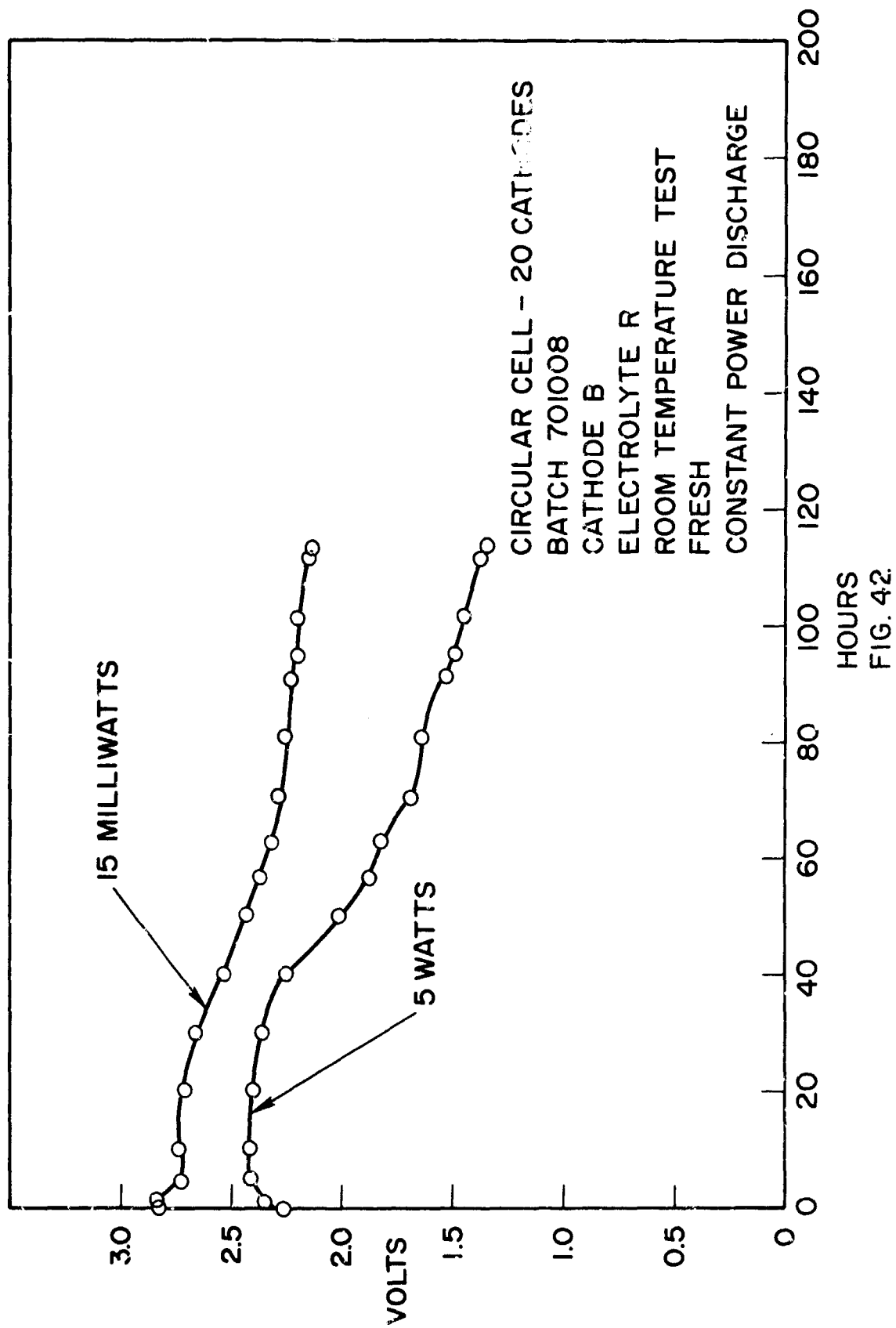
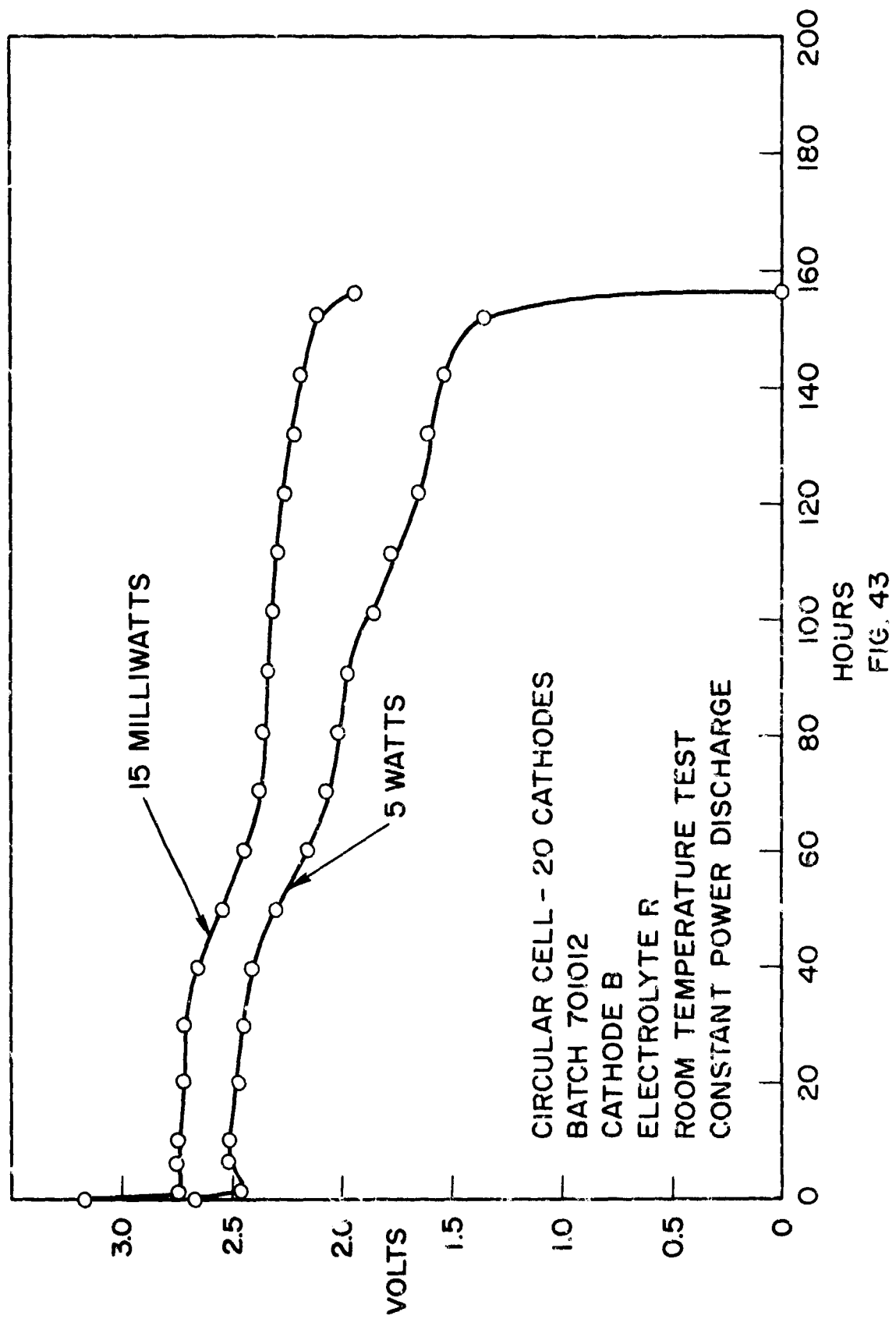


FIG. 40







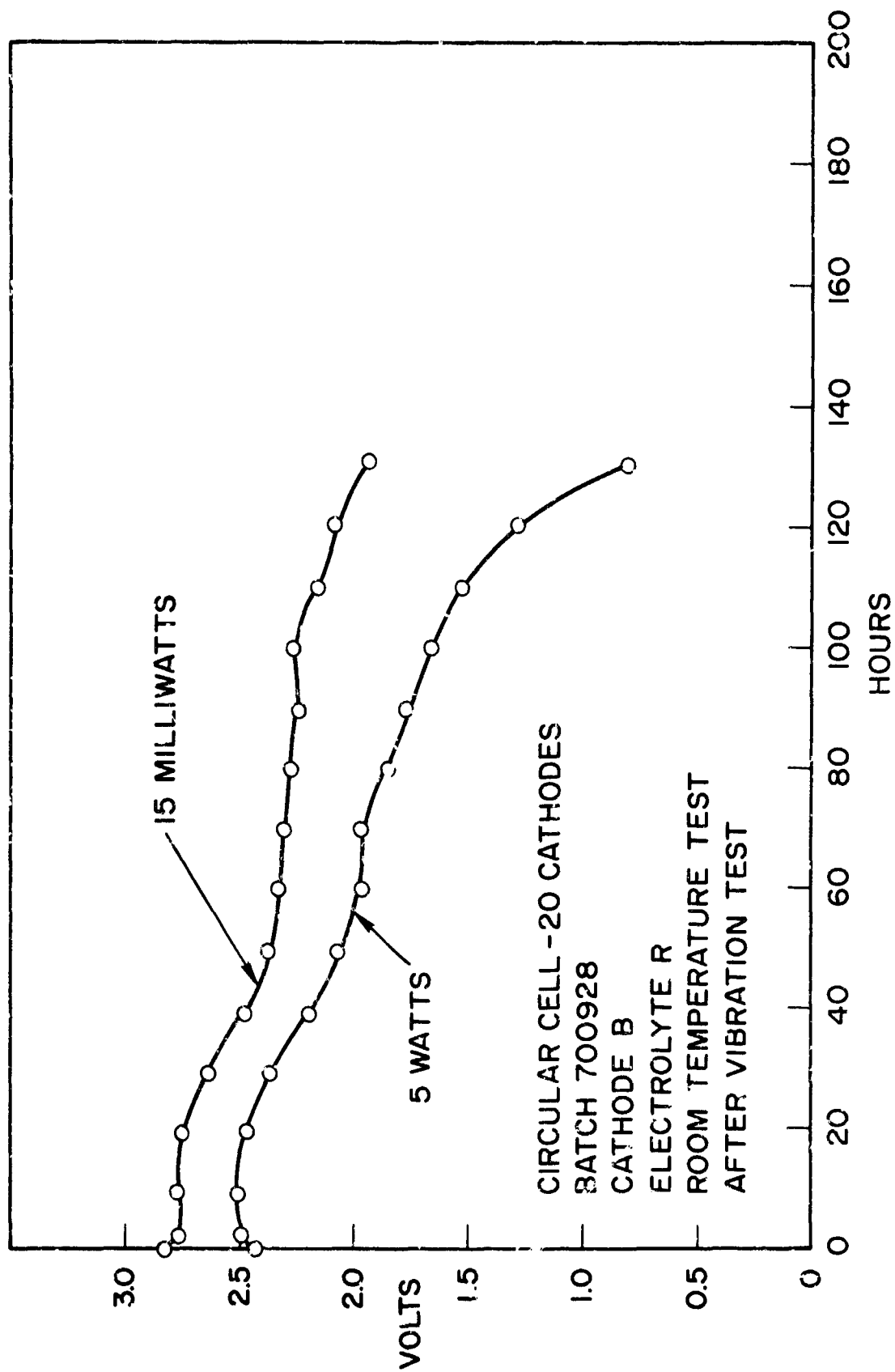
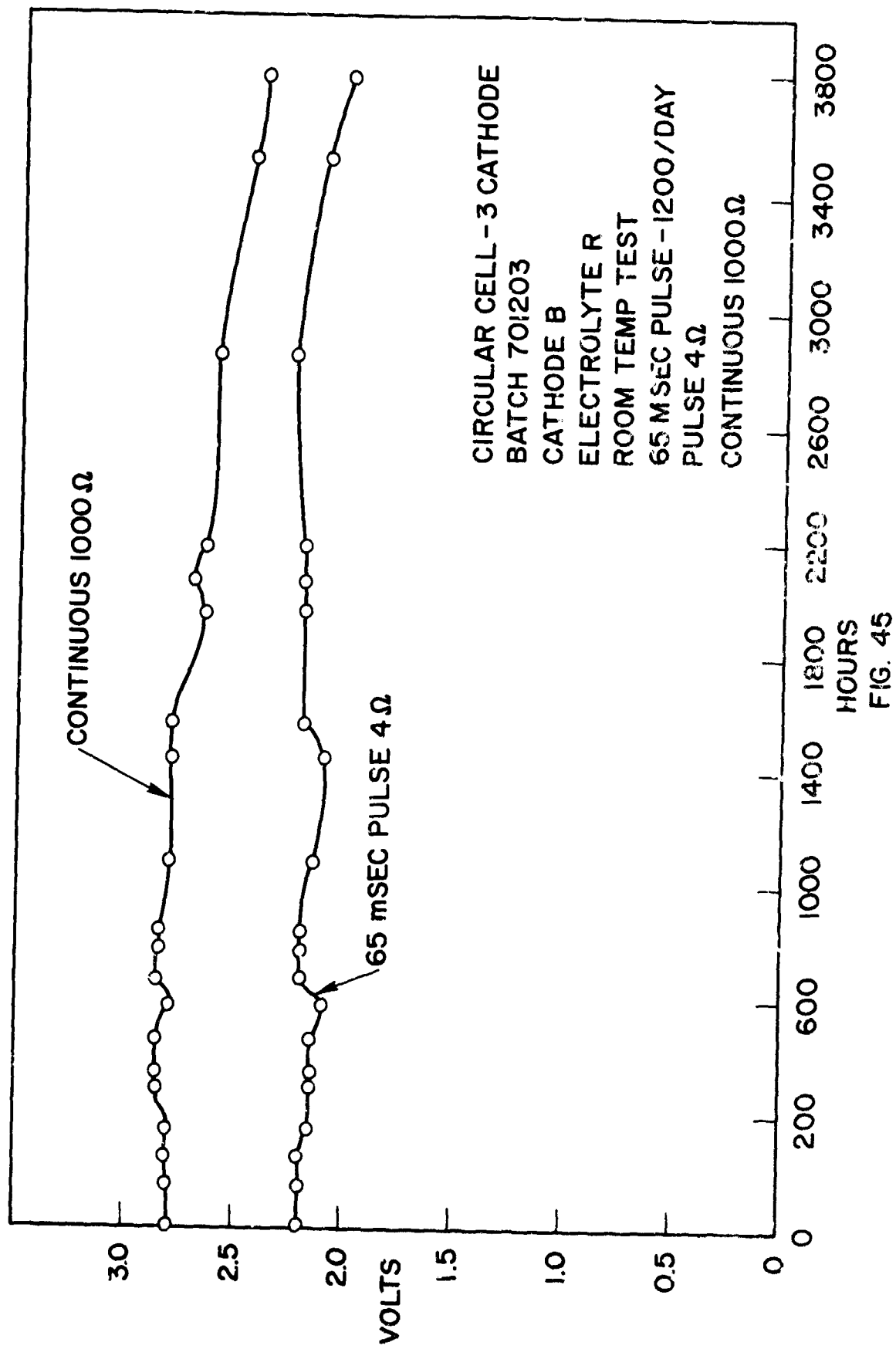
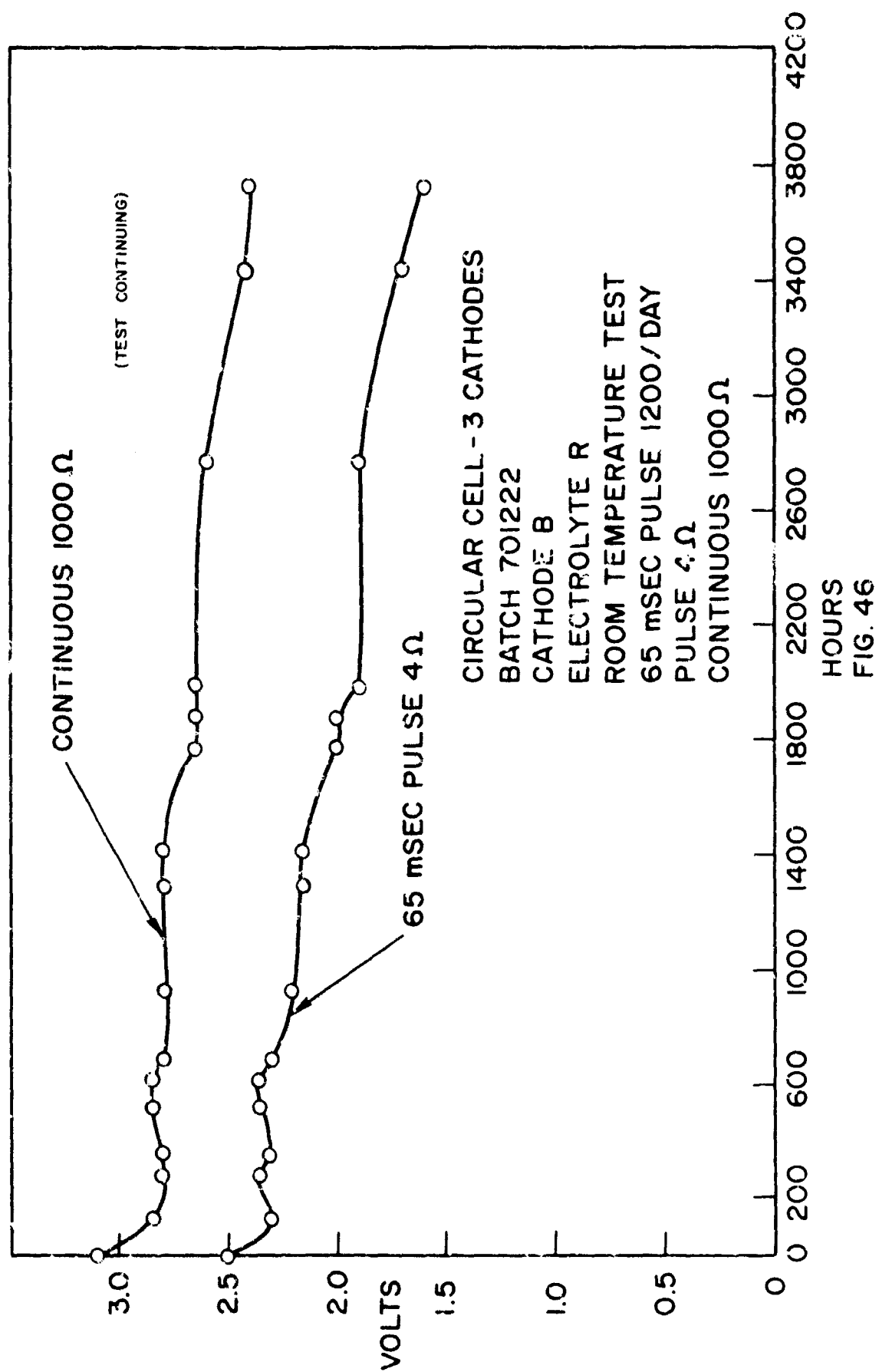
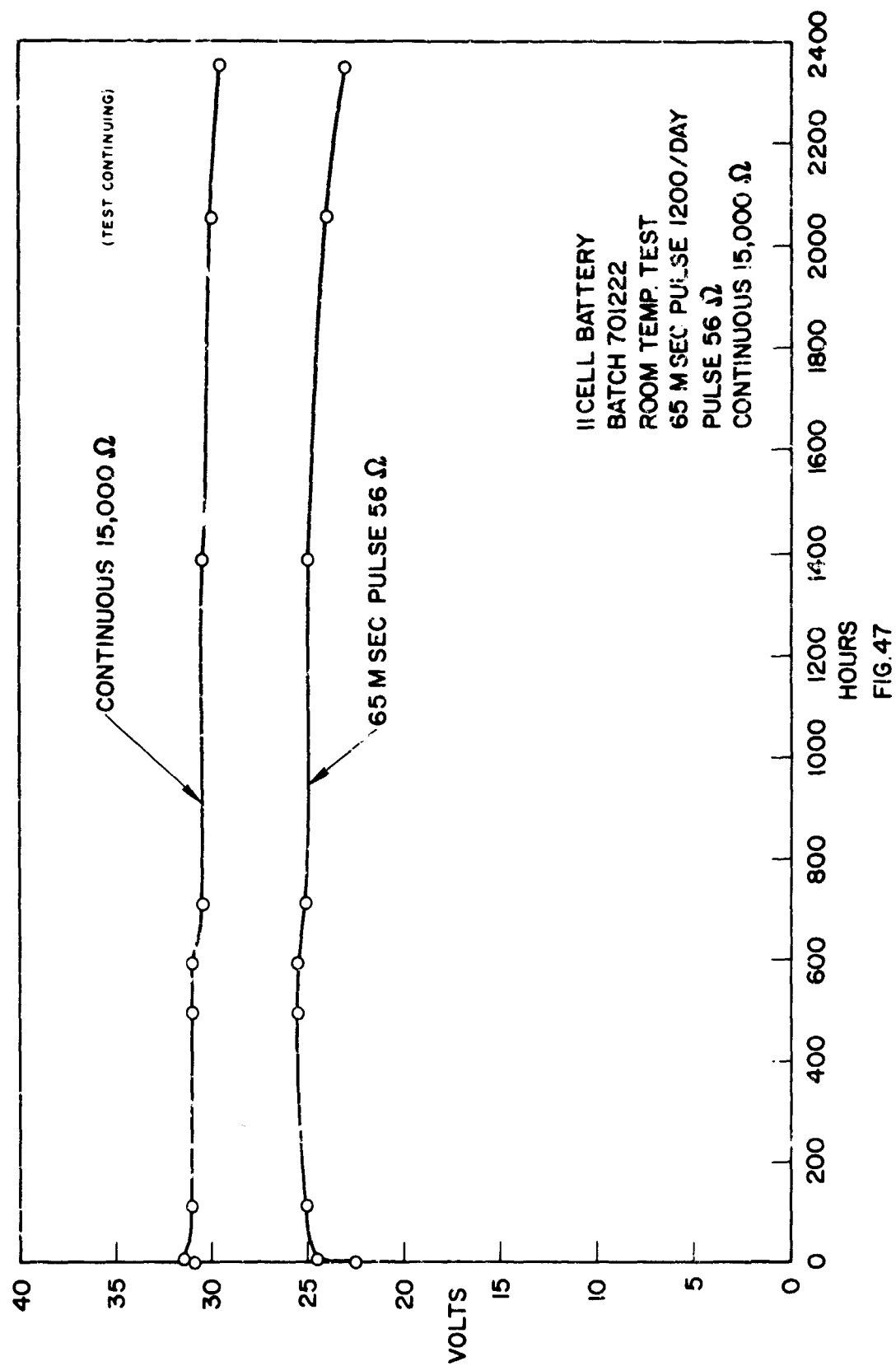
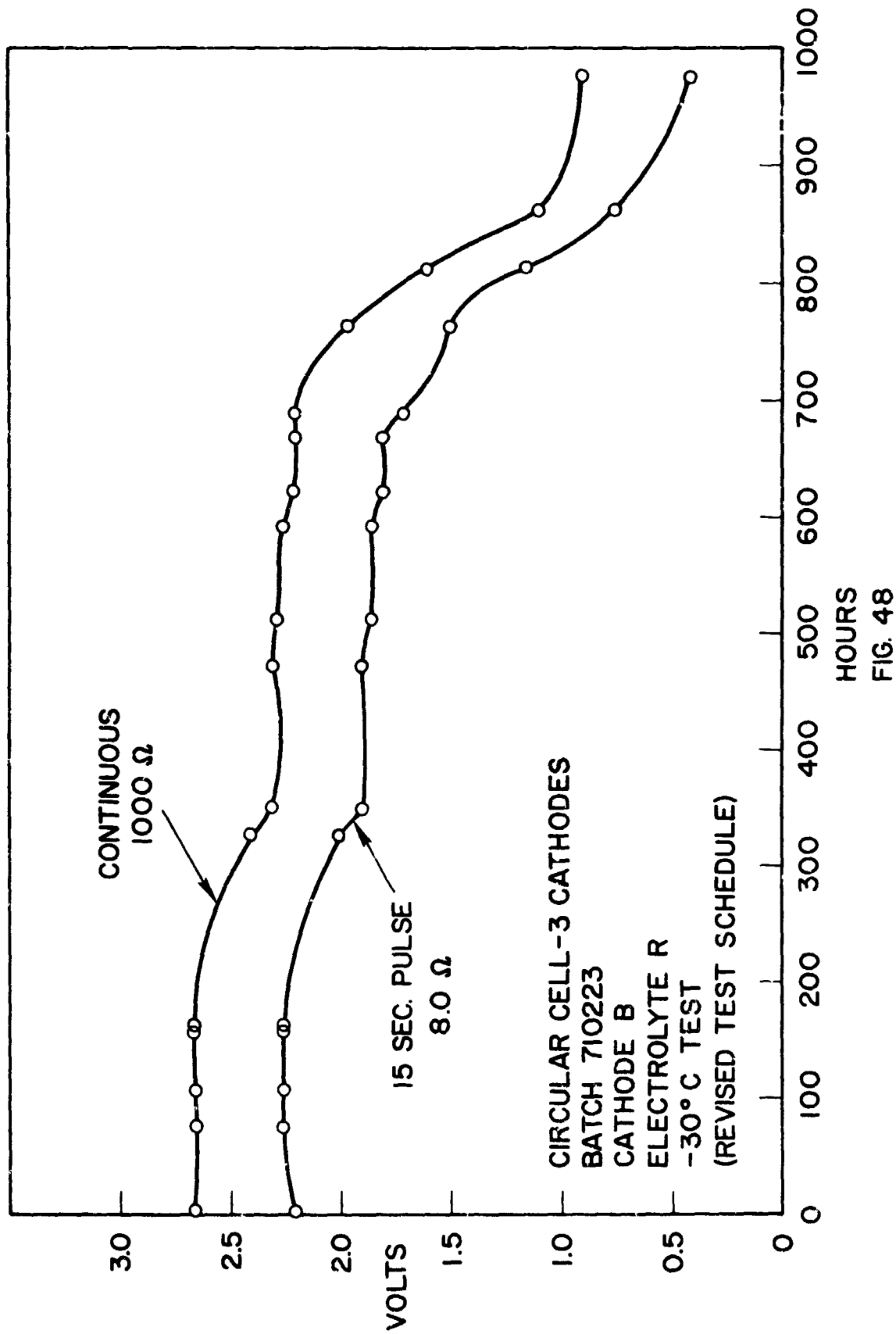


FIG. 44









APPENDIX IV

FAILURE ANALYSIS OF LB-35 CELLS

The cells delivered to Ft. Monmouth were our first generation prototype cells and some of them failed on room temperature storage or on various testing procedures. The failures were typified by low cell voltage, leakage, bulging of the can or corrosion.

The failed cells were returned to our laboratory and examined to determine the cause of the failures. The conclusions drawn from our examinations were the basis of a redesign of the heat seal used in subsequent cells that did not exhibit these failures. These conclusions are given below:

CONCLUSIONS OF EXAMINATIONS

Cell 1. This cell was pulse tested at room temperature and yielded 95 whr/lb. Internal examination indicated satisfactory construction, sealing and the expected discharge condition of the anodes. No failure was noted.

Cell 3. This cell failed on room temperature shelf test as a result of a leak in the foil bag at the final heat seal. The cell corrosion and discharge were a result of this leakage.

Cell 28. This cell failed on 130°F shelf test as a result of a leak at the final heat seal.

Cell 31. This cell failed on room temperature shelf test as a result of a pin hole leak in the foil bag.

Cell 32. This cell failed on low temperature pulse discharge test as a result of the passivating film on the anodes.

Cell 53. This cell failed on room temperature shelf test due to a high resistance short in the area where terminal tabs were heat sealed into the foil bag.

Cell 71. This cell failed on room temperature shelf test due to a low resistance short. The location of this short could not be determined.

Details of examination of these cells are given in Table 1 of this appendix.

TABLE 1.

Cell No.	Volts		OCV	Test Conditions	LPS Analysis 2-11-71
	11-24-70	1-4-71			
1	2.90	0		Room temp. discharge 95 whr/lb	OCV-2.2 volts. No corrosion exterior. Soldered during discharge tests. Potting in excellent condition. No internal leakage. Anodes nearly completely discharged. Two cathodes cracked--may be result of breaking potting while opening cell.
3	2.90	0		Room temp. shelf	OCV-0. Resistance 0.6 ohm. Slight corrosion at cold weld and at positive solder. Potting OK. Leak at top of foil bag--insufficient heat sealing. Electrolyte in can. Cell dry and anodes 98% discharged.
28	2.90	0		130°F storage	OCV-0. Resistance 20 ohm. No external corrosion. Can slightly bulged. Can was pressurized when opened. Electrolyte outside of foil bag. Positive terminal and glass to metal seal corroded. Foil bag insufficiently sealed at top. Potting good. Cell burned on opening. Anodes 95% consumed--further analysis impossible.
31	2.95	0		Room temp. shelf	OCV-0.01. Resistance 4.20. Corrosion on glass to metal seal. Liquid on top of cell. Can slightly bulged. Positive terminal corroded off internally. Pinhole leak found in foil bag. Potting good--heat seals good. Anodes 95% consumed. Cathodes firm --no cracks.
32	2.95	2.90		Pulse discharge at -30° below cutoff	OCV-2.65. Small amount of corrosion at positive terminal. Good potting. No signs of short. Anodes and cathodes good. Soldered connections good.

TABLE 1. (continued)

Cell No.	Volts		OCV	Test Conditions	LPS Analysis 2-11-71
	11-24-70	1-4-71			
53	2.8	0	Shelf room temp.	OCV-0. Resistance 0.7 ohm. Positive terminal solder gone. Corrosion and leakage at positive terminal. Potting not cured in top of can. Tabs has 2000 ohm between them when cut from cell terminals. Cell not shorted. Anodes discharged. No visible leaks in bag. Heat sealing good.	
71	2.85	0	Room temp. shelf	OCV-0. 0 ohm resistance. Can bulged slightly. Corrosion at cold weld. Pressure in can when opened. Potting OK. Liquid in cell bag. Anodes discharged, 95%, cell caught fire before examination was complete.	

SUMMARY OF EXAMINATION

The cell failures noted during this examination were all due to either electrolyte leakage or shorting. The final heat seal area was found to be unsatisfactory in nearly all of the failed cells. The cell terminal tabs were part of this heat seal area. The amount of heat applied to this seal was therefore critical in that too little heat resulted in a leak and too much heat resulted in a high resistance short on the cell.

The seal area was redesigned so that the cell tabs could be electrically tested and the seal leak tested prior to cell assembly. Cells were assembled using this improved sealing method that did not leak, short or show corrosion. The improved seals were not incorporated in the delivered end items due to schedule limitations.